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PRODUCTION OF TRITIUM BY CONTAINED
NUCLEAR EXPLOSIONS IN SALT:

I. LABORATORY STUDIES OF ISOTOPIC
EXCHANGE OF TRITIUM IN THE HYDROGEN-
WATER SYSTEM

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MASTER

OAK RIDGE NATIONAL LABORATORY

operated by

UNION CARBIDE CORPORATION

for the

U.S. ATOMIC ENERGY COMMISSION

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I. LABORATORY STUDIES OF ISOTOPIC EXCHANGE OF TRITIUM IN THE
HYDROGEN-WATER SYSTEM

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ABSTRACT

The results of all exchange experiments suggest that tritium produced by a contained nuclear explosion will be mixed with environmental water. Tritium exchange in the reaction $\text{HTO} + \text{H}_2 \rightleftharpoons \text{HT} + \text{H}_2\text{O}$ was catalyzed at 600°C by a number of oxygenated salts that occur in salt formations. Calcium sulfate was the best catalyst, with values as high as 49 and 77% with pure CaSO_4 and Drierite, respectively, in deep bed tests. Even under diffusion-controlled conditions, where reactant gases were passed over fine powders of CaSO_4 , exchanges were as high as 26% for residence times of 50 sec. Sodium chloride had no effect. At high temperatures in a plasma jet, with D_2 as a stand-in for T_2 , exchange was very rapid. For 90 msec residence time and a $\text{D}_2/\text{H}_2\text{O}$ feed ratio of 1.3, 53% of the water hydrogen exchanged with deuterium.

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1.0 INTRODUCTION

Laboratory work is presented on the isotopic exchange of hydrogen in the hydrogen-water system. This work was performed to determine the dilution of tritium produced by a contained nuclear explosion in bedded salt by hydrogen from environmental water since dilution of the tritium could interfere with its recovery. Of the several methods for separating hydrogen isotopes (1-3), probably none are economic for recovering tritium in concentrations of <10 ppm without an essentially infinite source of feed material.

Isotopic exchange between hydrogen and liquid water has been studied experimentally in connection with production of deuterium and heavy water (4). Only one study (5) has been made of rates above the critical temperature of water for the reaction $\text{HDO} \rightleftharpoons \text{H}_2\text{O} + \text{H}_2$, which was done in the temperature range 300-450°C with MgAl_2O_4 , MgCr_2O_4 , ZnCr_2O_4 , and ZnFe_2O_4 as catalysts. The major impurities in rock salt are anhydrite (CaSO_4), polyhalite ($\text{K}_2\text{SO}_4 \cdot \text{MgSO}_4 \cdot 2\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$), and silt. Isotopic exchange of hydrogen isotopes under ionizing radiation has been demonstrated (6,7).

Although the water content of rock salt is low, the lowest value that may reasonably be expected is 0.01% (8), for which value a $\text{H}_2\text{O}/\text{T}$ mole ratio of 2×10^3 is calculated* for the post-shot environment. Exchange of T to H_2O , if it occurred at all, would be practically 100% under these conditions, and the tritium concentration in the resulting steam would be only 100 ppm.

Another possible mechanism of tritium dilution is oxidation to water by oxygen-bearing impurities in the salt. This will be the subject of a later report. Dilution with hydrogen produced by dissociation of environmental water, although unlikely, is possible, and this will be studied if the first two mechanisms do not appear to hamper tritium production. Hydrolysis followed by isotopic exchange has not been considered since reactions with NaCl, though numerous, are of low yield.

*

The calculation assumed 100% efficiency of neutrons for tritium production and used published values (9,10) for the neutron flux of thermonuclear devices and the mass of molten salt per kiloton.

The reaction $\text{H}_2 + \text{HTO} \rightleftharpoons \text{HT} + \text{H}_2\text{O}$ was chosen for study since at tracer T concentrations it is the only reaction present. At higher T concentrations, as many as six species and many equilibria must be considered. However, the same general behavior would be expected for any of the other reactions toward different catalysts. For several of the reactions the calculated equilibrium exchange data (Table 1) show that the exchange is not statistical at lower temperatures and that the isotope effect is appreciable. Increasing the H_2O concentration in the reactant gases greatly increases the percentage exchange from the original molecule containing T.

Laboratory studies were made on the exchange of tritium in the 300-800°C range by conventional means with various compounds that occur in salt formations as catalysts. Since the degree of contacting of the reactants and products in the post-shot environment of an underground nuclear event is not known, the reaction was studied by two flow methods, one in which the contact between reactants and catalyst was good and another in which it was poor. In the dissociation temperature range, exchange was studied with a plasma jet, in which residence times are of the order of 10-100 msec, molecules are dissociated and atoms partially ionized, and the emerging plasma is quickly cooled. No attempt was made to separate the contributions to the total exchange by the various reactions between D_2 and H_2O .

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2.0 EXPERIMENTAL RESULTS

In a contained nuclear explosion, temperatures of several million degrees and pressures of several megabars are generated in a few microseconds. The temperature falls to the melting point of the containment medium in a few milliseconds since heat is rapidly transferred by radiation at these temperatures. The high shock pressure compresses the me-

Table 1. Calculated Effect of the H₂O Concentration in the Initial Gas on the Extent of Isotopic Exchange in the Isotopic Hydrogen-Water System^a

Reaction	Temp, °C	K	T Exchange from Original Molecule, %	
			50 mole % H ₂ O	91 mole % H ₂ O
$2T_2 + 2H_2O = HT + H_2 + HTO + T_2O$ $\Delta H_o^\circ = 975 \text{ cal}$	20	496	90.4	99
	200	75.7	85.5	99
	400	17.4	81.1	99
	800	6.61	74.9	99
$HT + H_2O = HTO + H_2$ $\Delta H_o^\circ = 1,221 \text{ cal}$	20	6.24	71.4	98.3
	200	2.66	62.0	96.3
	400	1.77	57.1	95.0
	800	1.19	52.2	92.6
$T_2 + H_2O = H_2 + T_2O$ $\Delta H_o^\circ = 2,465 \text{ cal}$	20	31.2	84.8	--
	200	6.04	71.1	--
	400	2.87	62.9	--
	800	1.85	57.6	--
$T_2 + H_2O = HT + HTO$ $\Delta H_o^\circ = -981 \text{ cal}$	20	15.9	80.0	--
	200	8.42	74.3	--
	400	6.19	71.2	--
	800	4.63	68.5	--

^a Calculated from data of Libby (11) and Jones (12) using reactant gases given on left side of equations.

dium as the shock wave moves outward from the center of detonation and creates a cavity in the medium. Thirty to 40% of the explosion energy is used in melting, and the molten material collects as a pool at the bottom of the cavity. Temperatures near the salt melting point are expected to persist for several months if the cavity remains intact, but will fall to near ambient in a few hundred milliseconds to several minutes if the cavity partially or completely collapses.

There are two discrete regions in the post-shot temperature-time history of a nuclear explosion contained in salt which could influence tritium exchange from T_2 to H_2O if the reactants are mixed and kinetics are favorable: the region from the final quench temperature up to the melting point of salt and the region of dissociation and ionization (above $10,000^\circ K$) for hydrogen and water molecules, which exist for only a few milliseconds. There is no reason to consider chemical reactions at temperatures above $10,000^\circ K$ since molecules are completely dissociated and atoms are in various degrees of ionization, creating an exchange path. However, when cooled to final conditions, the atoms and ions will recombine according to the thermodynamically preferred reactions if time and mixing of reactants are sufficient. For instance, at room temperature D_2 does not exchange a D atom for an H atom in H_2O in the absence of energy sources such as sufficient ionizing radiation or catalysts although the reaction is thermodynamically favorable. But when a mixture of D_2 and H_2O is heated to the dissociation region or beyond and then cooled to room temperature, the exchange will have occurred. The dissociation of the molecules furnishes the activation energy for the reaction. Since it is expected that the system will exist at temperatures at the melting point of salt ($804^\circ C$) or lower for a longer time than at higher temperatures, this region is probably the most important in defining most of the final chemical species.

2.1 Exchange with Flow through Packed Beds of Catalyst (Good Contact)

The reaction $HTO + H_2 \rightleftharpoons HT + H_2O$ was catalyzed by a number of solids that occur in salt formations, exchange being significant at $600^\circ C$. These results indicate that an isotope separation of tritium produced by an underground explosion is required since the tritium is diluted with environmental water.

All oxygenated salts and oxides tested in the form of large crystals were moderately effective catalysts (Table 2). The chlorides were not very effective, although calcium chloride was more effective than sodium. Potassium chloride, which is not likely to give results appreciably different from those with sodium chloride, was not tested. These results are consistent with the view that exchange involves one or both reactants on a surface that contains exposed oxygen atoms. In case of chlorides, exchange is believed to involve reversible hydrolysis of the salt, but in the experiment with calcium chloride the quartz was attacked, suggesting that the reaction is not entirely reversible.

Table 2. Exchange in the Reaction $\text{HTO} + \text{H}_2 \rightleftharpoons \text{HT} + \text{H}_2\text{O}$ with Various Catalysts

Temperature: 600°C

Catalyst bed: 1 in. dia, 6 in. deep

Flow rates, moles/min: $\text{H}_2 = 6.5 \times 10^{-3}$; $\text{H}_2\text{O} = 4.5 \times 10^{-4}$;
 $\text{HTO} = 4.1 \times 10^{-11}$; $\text{He} = 9.0 \times 10^{-3}$

Catalyst	Surface Area, m^2/g	% of Equilibrium Exchange ^a	Catalyst	Surface Area, m^2/g	% of Equilibrium Exchange ^a
NaCl	0.015	0.65	Al_2O_3	6.7	77
CaCl_2	0.39	6.5	K_2CO_3	0.073	80.5
K_2SO_4	0.007	42.5	CaSO_4^b	--	54.0
Silica gel	520	44	--	--	--

^a Equilibrium exchange is 90%.

^b Prepared by making plaster of Paris from reagent-grade $\text{CaSO}_4 \cdot 1/2 \text{H}_2\text{O}$, which was then calcined 8 hr at 900°C. The product was crushed before use but was not classified in mesh size. The particles were mainly large crystals.

For the flow rates used in these studies, increasing the bed length of Drierite from 3 to 12 in. did not appreciably alter the exchange (Table 3). The Drierite was always pretreated with hydrogen at temperature for several hours before use. Duplication of results was not always possible because of imperfect packing of the catalyst bed between experiments and attrition of particles during the heating and cooling cycles in the furnace.

Attempts to study the rate of exchange with different CaSO_4 powders (<325 mesh) as a function of surface area, flow rate, and temperature were unsuccessful (Table 4). The powders tended to behave as a mass of calcium

Table 3. Effect of Bed Depth on Exchange in the Reaction
 $\text{HTO} + \text{H}_2 \rightleftharpoons \text{HT} + \text{H}_2\text{O}$ over Fixed Drierite Beds

Temperature: 600°C

Bed dia: 1 in.

Initial flow rates, moles/min: $\text{H}_2 = 6.5 \times 10^{-3}$;
 $\text{H}_2\text{O} = 4.5 \times 10^{-4}$; $\text{HTO} = 4.1 \times 10^{-11}$; $\text{He} = 9.0 \times 10^{-3}$

Bed Depth, in.	Catalyst size ^a , mesh	Steady State Exchange Rate, moles ($\text{H}_2\text{O} + \text{HTO}$)/min	Mole % Exchanged at Steady State ^b
12	10-20	2.54×10^{-4}	56.5
	10-20	2.52×10^{-4}	56.0
	20-30	3.16×10^{-4}	70.2
	20-30	2.48×10^{-4}	55.0
6	20-30	3.23×10^{-4}	71.7
	20-30	3.23×10^{-4}	71.7
3	20-30	2.96×10^{-4}	65.8
	20-30	3.72×10^{-4}	82.8
	20-30	3.84×10^{-4}	85.5
	20-30	3.37×10^{-4}	74.9

^a For 20-30 mesh material the surface area is 9.7 m²/g; 10-20 mesh was not measured.

^b At 600°C, calculated equilibrium exchange is 90%.

Table 4. Effect of Temperature on Exchange in the Reaction
 $\text{HTO} + \text{H}_2 \rightleftharpoons \text{HT} + \text{H}_2\text{O}$ over Finely Powdered CaSO_4

Bed dia: 1 in.

Flow rates, moles/min: $\text{H}_2 = 6.5 \times 10^{-3}$; $\text{H}_2\text{O} = 4.5 \times 10^{-4}$;
 $\text{HTO} = 4.1 \times 10^{-11}$; $\text{He} = 9.0 \times 10^{-3}$

Temperature, °C	Surface Area, m ² /g	Bed Depth, in.	% of Equilibrium Exchange ^a
600	1.3	7/16	0
	1.3	7/16	0
	10.0	1/4	26
	10.0	3/32	5.7
	0.82	3/32	7.8
700	1.3	7/16	0
	10.0	3/32	19.8

^a Under conditions of these experiments equilibrium exchange is 90% at 600°C and 93% at 700°C.

sulfate, as evidenced by the fact that upflow of gaseous reactants caused the mass to float upward in the furnace tube. The different catalytic activities of different preparations of calcium sulfate are probably due to differences in packing properties.

2.2 Exchange with Flow Over Layers of Catalyst (Poor Contact)

Tritium exchange was diffusion controlled, as shown by the rate of exchange when a gaseous mixture of $\text{HTO} + \text{H}_2 + \text{H}_2\text{O}$ was passed over layers of CaSO_4 (Table 5). Equilibrium exchange was not reached (Fig. 1).

Table 5. Exchange for the Reaction $\text{HTO} + \text{H}_2 \rightleftharpoons \text{HT} + \text{H}_2\text{O}$ with CaSO_4 Catalyst

Initial flow rates, moles/min: $\text{H}_2 = 6.5 \times 10^{-3}$; $\text{H}_2\text{O} = 4.5 \times 10^{-4}$;
 $\text{HTO} = 4.1 \times 10^{-11}$; $\text{He} = 9.0 \times 10^{-3}$

Temp, °C	Wt of CaSO_4 , g	Surface Area, m^2/g	Furnace Tube Vol ^a , cc	Steady State Ex- change, moles ($\text{H}_2\text{O} + \text{HTO}$)/min	Mole % Exchanged at Steady State ^b
380	3.03	0.82	315	0.93×10^{-5}	2.07
600	1.00	0.82	35	3.14×10^{-5}	6.98
		0.82	35	3.23×10^{-5}	7.18
		10.0	35	2.65×10^{-5}	5.89
		10.0	35	2.65×10^{-5}	5.89
	3.03	0.82	35	6.01×10^{-5}	13.35
		10.0	35	5.13×10^{-5}	11.40
		0.82	315	6.11×10^{-5}	13.55
		10.0	315	7.30×10^{-5}	16.22
	6.03	10.0	35	7.38×10^{-5}	16.40
		10.0	315	10.52×10^{-5}	23.38
700	0.25	0.82	315	8.84×10^{-5}	19.64
	12.03	10.0	315	11.59×10^{-5}	25.75
	1.00	0.82	35	5.75×10^{-5}	12.78
		10.0	35	5.88×10^{-5}	13.07
		0.82	315	9.82×10^{-5}	21.82
	3.03	0.82	315	9.82×10^{-5}	21.82

^a 35-cc tube, 2.0 cm i.d.; 315-cc tube, 4.0 cm i.d.

The exchange increased with increasing temperature and was practically independent of CaSO_4 surface area and reaction tube volume. The apparent increase of exchange with weight of CaSO_4 is not real and was shown to be due to decreasing the distance between the reactant gas entry point and the surface of the CaSO_4 layer (Fig. 2, Table 6). Decreasing

Fig. 1. Calculated equilibrium exchange in the reaction $\text{HTO} + \text{H}_2 \rightleftharpoons \text{HT} + \text{H}_2\text{O}$. Initial conditions: H_2O 4.5×10^{-4} mole, H_2 6.5×10^{-3} mole, HTO 4.1×10^{-11} mole. $\Delta H = 1,221$ cal. Calculated from data of Libby (10) and Jones (11).

Fig. 2. Apparent effect of weight of CaSO_4 on tritium exchange in the reaction $\text{HTO} + \text{H}_2 \rightleftharpoons \text{HT} + \text{H}_2\text{O}$. Temperature 600°C ; surface area $10.0 \text{ m}^2/\text{g}$; flow rates, moles/min: H_2 6.5×10^{-3} ; H_2O 4.5×10^{-4} ; HTO 4.1×10^{-11} ; He 9.0×10^{-3} .

Table 6. Effect of Distance from Entrance Gas Line to Edge of Sample

Initial flow rates, moles/min: $H_2 = 6.5 \times 10^{-3}$; $HTO = 4.1 \times 10^{-11}$;
 $H_2O = 4.5 \times 10^{-4}$; $He = 9.0$
 Surface area: $10.0 \text{ m}^2/\text{g}$

Distance from Gas Entry Tube to Sample, cm	Wt of $CaSO_4$, g	Steady State Ex- change, moles ($HTO + H_2O$)/min	Mole % Exchanged at Steady State
3.0	6.03	5.39×10^{-5}	12.0
3.0	3.03	5.13×10^{-5}	11.4
1.3	6.03	7.38×10^{-5}	16.4
4.0	1.00	2.65×10^{-5}	5.9

the flow rate of reactant gases by a factor of 2 increased the net exchange by a factor of 1.5, but further decrease of flow rate caused no additional increase (Table 7). The apparent activation energy calculated from the Arrhenius equation is $8.8 \pm 1.0 \text{ kcal}$ (Fig. 3). Sodium chloride did not catalyze the reaction under these conditions. Anhydrite samples from the Gnome site were only 25% as effective as was pure $CaSO_4$ under the same test conditions. At 700°C traces of H_2S were produced, which showed that very slight reaction was occurring between H_2 and $CaSO_4$; thus the catalyst at this temperature contained traces of CaS and CaO .

Table 7. Effect of Flow Rate on Exchange in the Reaction

Wt of $CaSO_4$ ($0.82 \text{ m}^2/\text{g}$): 3.03 g

Temperature: 600°C

Furnace tube volume: 35 cc

Flow Rate, moles/min					Steady State T Exchange	
H_2	He	HTO	H_2O	Total Gas	moles(H_2O+HT)/ min	% T Exchange
6.5	8.50	4.1×10^{-8}	0.45	15.45	6.01×10^{-5}	13.3
3.54	4.00	2.1×10^{-8}	0.214	7.75	4.40×10^{-5}	20.5
1.75	2.05	1.0×10^{-8}	0.111	3.91	2.23×10^{-5}	20.2

The data show that $CaSO_4$ provides a path whereby the exchange can occur. Residence times of 5-50 sec over the catalyst produced 6-26% exchange of T at 600°C . Even under diffusion-controlled conditions, it

Fig. 3. Effect of temperature on T exchange in the reaction $\text{HTO} + \text{H}_2 \rightarrow \text{H}_2\text{O} + \text{HT}$. CaSO_4 (0.82 m²/g), 3.03 g. Flow rates, moles/min: H_2 6.5×10^{-3} ; H_2O 4.5×10^{-4} ; HTO 4.1×10^{-11} ; He 9.0×10^{-3} .

would be expected that the T produced by a nuclear detonation in salt would exchange appreciably with environmental water if left in the medium for a few hours.

2.3 Exchange of D₂ with H₂O in a Plasma Jet

The results of D₂ and H₂O exchange in a plasma jet show that if T₂ and H₂O are mixed within the fireball of the exploding device, dilution of tritium by isotopic exchange will be severe.

Table 8. D₂ Exchange with H₂O in a Plasma Jet

Temperature: 10,000-15,000°K

Low-momentum nozzle, 500 amp at 32.5 volts

Flow rates, liters/min: A = 4.7; H₂ = 0.9

Gas mixture residence time in flame: ~90 msec

D ₂ /H ₂ O Mole Ratio	Atom % D in water after Exchange	g of D ₂ O/g of Solution after Exchange
11	82.6	0.775
2.3	49.1	0.517
2.3	59.0	0.616
2.3	51.7	0.543

3.0 EXPERIMENTAL METHODS

3.1 Tritium Exchange Experiments

Ordinary hydrogen and helium were mixed in a Y tube at selected flow rates and saturated in a bubbler tube with tritiated water (specific activity 147 mc/ml) in a constant temperature bath at $25.00 \pm 0.05^{\circ}\text{C}$ to give a water concentration of 23 mg/liter of gas (Fig. 4). For the experiments in which contact of catalyst and reactant gases was good the gas flowed through the catalyst bed (Fig. 5a), but for those with poor contact the gas flowed over the catalyst (Fig. 5b).

The gas stream was allowed to flow through the furnace tube, after which water was removed by a tared Drierite trap and the hydrogen was converted to water by a 12-in.-deep bed of CuO at 380°C . The resulting water was analyzed for tritium content by liquid scintillation counting. Steady-state exchange was reached in 90-120 min operation of the apparatus. Exchange percentages and rates were reported at steady state. A blank correction was made for the hot quartz furnace tube.

All chemicals used were reagent grade. Calcium sulfate catalysts were prepared by heating $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ to 900°C in a muffle furnace and then calcining at 900°C for 12-20 hr.

3.2 Plasma Jet Experiments

The apparatus for this study (Figs. 6-8) was a Thermal Dynamics Model F-40 plasma jet unit with a low-momentum nozzle. Temperatures of argon plasma produced by this unit have been estimated at 10,000-15,000 $^{\circ}\text{K}$ by spectroscopic measurement of line broadening. Deuterium gas is saturated with ordinary water to give the desired $\text{D}_2/\text{H}_2\text{O}$ ratio and injected into the jet nozzle at 900 cc/min, where it mixes with the argon plasma. The plasma is cooled by the water-jacketed brass chamber, and water is condensed by a cold trap. The D content of the condensed water is determined mass spectrographically.

Fig. 4. Schematic diagram of exchange apparatus.

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Fig. 5. Typical furnace tube for (a) fixed bed experiments and (b) experiments with poor gas-solid contact.

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Fig. 6. Schematic of D₂-H₂O exchange experiments with a plasma jet.

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Fig. 7. Plasma torch and water-jacketed cooling chamber.

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Fig. 8. Control console for plasma jet.

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