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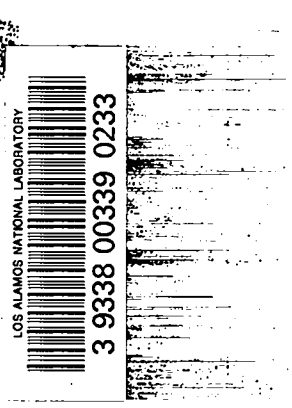

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August 8, 1950 LA-1138

THE REACTION OF TRITIUM AND STOPCOCK GREASE

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THE REACTION OF TRITIUM AND STOPCOCK GREASE

I. Introduction:

This investigation was begun as a result of a mass spectrometric measurement of two samples of tritium which had been stored for several months in pyrex glass bulbs using stopcocks. The first of these was originally supposed to be of extremely high isotopic and chemical purity. What was observed at this time was that 1.4% H had accumulated, and that tritiated methane, CT_4, was also present to the extent of 0.18%. A consistent attempt had been made in this sample to protect the gas from contact with the grease by covering the stopcock with mercury. The second sample was a composite of T_2 samples of various degrees of enrichment with an isotopic analysis of 92% T. No protection had been attempted during its storage, and analysis showed 0.86% CT_4 to be present.

II. Net Tritium Degradation:

In order to measure the rate of this effect two samples, I and II of chemically pure 92% tritium were placed at 40 cm pressure in a pair of 5 cc bulbs equipped with stopcocks and allowed to stand. A third bulb III was smeared with grease over a large area in addition to the stopcock in order to enhance the effect.

The results are contained in Table I.

Table I. Atomic percent T in H_2, HT, T_2

Bulb	7/11	7/13	7/18	Rate per day
I	91.6	90.4	88.8	-0.54%
II	91.6	90.1	87.2	
III	91.6	88.3	82.0	-1.40%

Thus even in an unprotected bulb of small volume it appears safe to handle these gases in systems equipped with stopcocks for times of less than, say, an hour, but for long term storage such contact is definitely deleterious.

However the methane appearance in these samples proved to be negligible! In the whole time none of them exhibited more than 0.01% methane. Up to 0.4% N_2 (in sample III) appeared either through leakage or from release from the grease. Comparable quantities of CO_2 were similarly observed. These data are presented in Table II. These results suggest no obvious interpretation. The very large CO_2 in comparison to the N_2 eliminates the possibility of simple combustion of the atmospheric oxygen carried in with the indicated nitrogen. The original samples referred to in the introduction did not show significant N_2 or CO_2. Such results are very

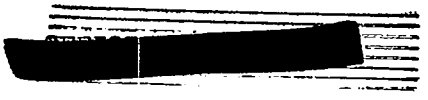

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Table II. Final Impurity Analysis -

Percent Impurity Accumulated

Bulb	ΔCT_4	ΔN_2	ΔCO_2
I	.010	.010	.026
II	.005	.119	.171
III	.005	.380	.314

unsatisfactory, of course, and must await a further study for fuller explanation.

Two other bits of information have been secured. On July 1 a run was attempted with chemically purified tritium but was inadvertently contaminated with air while manipulating it with the resulting analysis shown in Table III.

Table III. Run with Air-contaminated Tritium

Date	Time	N_2	O_2	CO_2	O_2-Deficiency
7/1	5 pm	8.81	2.23	0.01	0.02
7/3	8 am	13.51	0.89	0.68	2.49

A second such test occurred with some of the methane contaminated gas in which air appeared accidentally with the following results.

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Date	Time	N_2	O_2	CO_2	CT_4	O_2-Deficiency
6/29	12 M	0.63	0.08	0.16	0.86	0.08
6/29	4 pm	7.11	1.41	0.29	0.71	0.37
6/30	8 am	8.39	0.72	0.50	0.65	1.38

One may say from these data that oxygen is sufficiently reactive in the presence of tritium to oxidize both grease and any methane present in the gas phase. There is no indication here but that there is sufficient oxygen in the admitted air to account for all the CO_2 formed, in contrast to the data of Table II. The balance is sufficiently close however that not much reaction can be expected to have occurred between the tritium and oxygen directly to form water.

A final test was made by admitting tritium to the spectrometer manifold and sparking the glass with a Tesla coil leak tester for ten minutes, which resulted in copious liberation of methane and extensive reduction in the isotopic enrichment.

	Methanes	percent T
Before	0.01	91.4
After	2.9	60.9

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Since such slight production of methane occurred on standing alone it is concluded that probably the methane which originally started this investigation occurred in the above manner.

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