



## Experimental

All chemicals were obtained commercially. Radiochemical purity was recorded on Radio-Gas Chromatography, Model SQ-203. Tritium was counted using Liquid Scintillation Counter, Model FJ-353G.

### (1) Synthesis of crotyl alcohol 1

A 500 ml, three-necked, round-bottomed flask, equipped with an efficient stirrer and a reflux condenser attached to a device for measuring gas evolution was charged with 6 g (0.16 Mole) of  $\text{LiAlH}_4$  and 200 ml of anhydrous ether. 25 ml (0.30 Mole) of crotonaldehyde was dissolved in 50 ml of anhydrous ether and added dropwise with stirring to the lithium aluminum hydride solution. When the addition was complete, the mixture was refluxed for overnight. Excess of lithium aluminum hydride was decomposed by careful addition of 40 ml of water. The ether layer was collected and the aqueous layer was separated, which was extracted three times with 20 ml portions of ether. The ether layer and ether extract were combined and washed with water. Ether was removed by distillation, then at 118 °C 18.6 g (yield 86%) of the crotyl alcohol was collected.

Anal. Calcd. for  $\text{C}_4\text{H}_8\text{O}$ ; C, 66.67; H, 11.11. Found; C, 66.65; H, 11.10.

### (2) Synthesis of [2,3- $^3\text{H}$ ]-n-butyl alcohol 2

0.1 ml of crotyl alcohol and 1.0 ml of acetone were added to a 25 ml of reaction flask fitted with a magnetic stirring bar and 50 mg of 10% Pd/C catalyst. It was then attached to the tritiation system. The flask was cooled with liquid nitrogen, and evacuated to 0.13 Pa. At the same time, 87 Pa of tritium (purity >99%) was introduced to the flask. After the reaction mixture was thawed, the crotyl alcohol and tritium gas were stirred for 1.5 hours over the 10% Pd/C. The flask was refrozen with liquid nitrogen and unreacted tritium was removed to a tritium storage tank. The catalyst was removed by filtration and the product, [2,3- $^3\text{H}$ ]-n-butyl alcohol (2.5TBq yield 99%), was collected. The product needed no further purification and was submitted to determine its radiochemical purity by Radio-Gas Chromatography and for tritium counting in Liquid Scintillation Counter.

## Results and discussion

**Synthesis of crotyl alcohol** The rate of adding must be controlled at such a rate that the liquid boiled gently to prevent side reaction that was unnecessary. The significant characteristic of our experiment is that the product, [2,3- $^3\text{H}$ ]-n-butyl alcohol does not need to be purified, but its radiochemical purity is very high (99%). It has been further proved that crotyl alcohol synthesis in our laboratory is very stable to radiation and not decomposed after tritiation.

**Synthesis of [2,3-<sup>3</sup>H]-n-butyl alcohol** The reaction time, the quantity of solvent and the quantity of catalyst were important factors to the synthesis of [2,3-<sup>3</sup>H]-n-butyl alcohol. If the reaction time was less than 70 minutes, crotyl alcohol could not be reduced completely to n-butyl alcohol [see Table 1 ( in this experiment, we used hydrogen instead of tritium to get all the data, because the property of hydrogen is same as tritium. )]. So the reaction time should be longer than 70 minutes at this reaction system.

Table 1 Relation of reaction time on amount of hydrogen absorbed

Reaction time (min.)	Crotyl alcohol (mg)	6% Pd/C (mg)	Amount of hydrogen absorbed (kPa)
10	500	500	52
20	500	500	91
30	500	500	120
40	500	500	145
50	500	500	166
60	500	500	179
70	500	500	190
80	500	500	190

The quantity of solvent was an important factor to hydrogen addition to crotyl alcohol (see Table 2 ). If the volume ratio of solvent to crotyl alcohol was smaller than 4:1, the time needed for reducing completely crotyl alcohol to n-butyl alcohol would be longer. So the volume ratio of solvent to the crotyl alcohol must be over 6:1. Also the quantity of catalyst could influence the rate of reaction for

Table 2 Effect of quantity of solvent on the balanced time

Quantity of solvent (ml)	6%Pd/C (mg)	Crotyl alcohol ( $\mu$ l)	Balanced time (min.)
1	500	500	270
2	500	500	130
3	500	500	90
4	500	500	80
5	500	500	70

introduction of hydrogen into the crotyl alcohol (see Table 3). If the ratio of quantity of catalyst to crotyl alcohol from 1:5 to 1:1.7, the rate of reaction was increased over two times. So the quantity of catalyst to crotyl alcohol should be selected over 1. According to the above factors, 50 mg of

Table 3 Effect of quantity of catalyst on the balanced time

6%Pd/C catalyst (mg)	Crotyl alcohol (mg)	Quantity of solvent (ml)	Balanced time (min.)
100	500	5	190
200	500	5	90
300	500	5	80
500	500	5	70

10% Pd/C, 1.0 ml of solvent, 0.1 ml of crotyl alcohol and 1.5 hours of reaction time were selected as the optimum tritiation condition.

#### Refereces

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