the reversibility of the reaction with lipoic acid as substrate. These results are in conformity with those of Gunsalus[3] and Goldman[6] and in partial agreement with those of Sanadi and co-workers.[4,5]

Results of the stoichiometric experiments are shown in Table II. Since even by coupling with lactic dehydrogenase or alcohol dehydrogenase not more than 50% of the (±)-dihydrolipoic acid was oxidized, the stereospecificity of the enzyme was suspected. (−)-Dihydrolipoic acid was not fully oxidized, however, which may be due to contamination with the other isomer.

TABLE II

Substrate	μM. added	μM. oxidized
(±)-Dihydrolipoic acid	2.0	0.9
(−)-Dihydrolipoic acid	2.0	1.7

The incubation was carried out at 25° for 30 min. in 1.5 ml. containing 60 μM TRIS (pH 8.0), 5 μM. DPN, 2 μM. of substrate and 39 μg. of dihydrolipoic dehydrogenase. Aliquots were assayed for SH groups[11]

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(11) H. Beinert, unpublished method.

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THE POLYBORANE CARBONYL B₄H₈CO

Sir:

When borine carbonyl (BH₃CO) was discovered, a less volatile carbonyl also was observed.[1] Its formula now has been indicated by the virtually quantitative synthesis B₅H₁₁ + 2CO → B₄H₈CO + BH₃CO, and confirmed by analysis. This complete formation of B₄H₈CO and BH₃CO strongly suggests that the most typical reactions of B₅H₁₁ begin with cleavage into B₄H₈ and BH₃ fragments. The removal of one BH₃ from an open corner of the B₅H₁₁ structure[2] would leave B₄H₈ having the shape of a B₄H₁₀ molecule[2] with two hydrogen atoms missing. This pattern might help to explain why B₅H₁₁ so readily absorbs hydrogen to form B₄H₁₀ and B₂H₆.[3]

Synthesis.—The reactions summarized in Table I occurred in sealed 50-ml. bomb tubes at 22–27°. In Expts. 3 and 4 all CO was accounted for before and after the reaction; and CuO combustion demonstrated the absence of hydrogen. The B₅H₁₁

(1) A. B. Burg and H. I. Schlesinger, THIS JOURNAL, **59**, 780 (1937).

(2) W. N. Lipscomb, *J. Chem. Phys.*, **22**, 985 (1954).

(3) A. B. Burg and H. I. Schlesinger, THIS JOURNAL, **55**, 4009 (1933).

had been purified by thorough fractionation (micro-column with −78° reflux); vapor tension 52.8 mm. at 0° and m.p. −123.1°, showing the absence of B₅H₉.[3,4] Its gas-volume may have been slightly underestimated. The B₄H₈CO also was reflux-fractionated, delivering only slight traces of more and less volatile material.

TABLE I

SYNTHESIS OF B₄H₈CO

Expt. No.	B₅H₁₁ (mmoles)	Pressure of CO (atm.)	Time at 22–27° (hr.)	B₄H₈CO (mmoles)	BH₃CO (mmoles)	CO Consumed (mmoles)
1	3.7	7.0	4.0	1.6	3.4	..
2	0.72	19.5	16.0	0.54	0.61	..
3	1.35	19.8	1.92	1.32	1.36	2.6
4	1.08	20.0	2.50	1.07	1.15	2.3

The long-run Expts. 1 and 2 gave much non-volatile solid. The trace of such material from Expt. 4 was dissolved in nitric acid and the boron determined as 0.067 mmole (1.4%).

Physical Properties.—The gas-phase mol. wt. of B₄H₈CO was 77.9 or 79.4 (calcd., 79.35). Its vapor tensions (Table II) gave the equation log p_{mm} = 4.555 − 0.00340T + 1.75 log T − 1649/T; b.p. 59.6°; Trouton constant, 21.0 cal./deg. mole. Crystallization difficulties sometimes prevented m.p. determinations; however, two apparently reliable results (Stock plunger method) were −114.4 and −114.6°

TABLE II

VAPOR TENSIONS OF B₄H₈CO

t, °C.	−76.3	−72.1	−58.8	−43.1	−37.0	0.00
p_{mm} (obsd.)	0.34	0.49	1.61	5.47	8.5	71.2
p_{mm} (calcd.)	0.33	0.50	1.64	5.49	8.4	71.3

Analyses.—We found no single quantitative reaction that would give a complete elementary analysis, but separate determinations, based upon different reactions, verified the formula B₄H₈CO. A 60% dissociation at room temperature gave 96% of the expected CO, but the remaining sample was uncertain. Complete decomposition (Vycor tube, 800–900°) gave 81% of the expected CO, raised to 85% by pure-O₂ combustion of the non-volatile residue. Boron was estimated as 4.04 B per molecule, but hydrogen ran low. Bromine oxidation at 100° gave 0.81CO, 8.03 (± 0.07) HBr and 3.63 BBr₃ per mole; then nitric-acid oxidation of the residue brought B to 3.96 (± 0.17) per mole. Thus the formula B₄H₈CO seems reliable.

Chemical Behavior.—B₄H₈CO reacts with H₂O or (CH₃)₃N, liberating virtually no carbon monoxide. Further studies are in progress.

(4) A. Stock and W. Mathing, *Ber.*, **69B**, 1464 (1936).

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