Table 4. Mass Spectrographic Analyses of Reaction Gases a,b,c and Wet-Chemical Analyses of Residual CO₂ in Unreacted D°Lomite: Dolomite-Hydrogen System

| Experiment No. | 39 | 41 | 42 | 43 | 44 | 45 | 46 | 47 | 49 | 50 | 51 | 56 | 57 | 58 | 59 | 83 | 84 |
|---|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| Temperature (°C) | 520 | 525 | 550 | 620 | 735 | 620 | 620 | 620 | 735 | 735 | 735 | 735 | 735 | 735 | 835 | 605 | 605 |
| Pressure (psi) | 5,000 | 5,000 | 5,000 | 5,000 | 5,000 | 5,000 | 5,000 | 5,000 | 5,000 | 5,000 | 5,000 | 5,000 | 4,000 | 4,000 | 5,000 | 2,000 | 2,000 |
| Wt. % CO2 remaining in solid | N.A.d | 44.3 | 40.2 | 25.6 | 10.1 | 25.4 | 30.7 | 19.4 | N.A. | N.A. | N.A. | N.A. | N.A. | N.A. | 0.87 | N.A. | N.A. |
| Duration of run (hours) | 4 | 4 | 4 | 4 | 4 | 8 | 2 | 12 | 4 | 4 | 4 | 4 | 6 | 7 | 4 | 2 | 4 |
| Mole % CH ₄ in gas | 0.01 | 0.02 | 0.04 | 1.20 | 2.34 | 1.82 | 1.53 | 2.05 | 2.00 | 1.88 | 1.77 | 1.80 | 2.86 | 4.76 | 2.12 | 0.46 | 2.64 |
| Mole % C ₂ H ₆ in gas | | | 0.01 | 0.02 | _ | 0.04 | 0.05 | 0.04 | - | - | = | - | - | 0.03 | | 0.03 | 0.14 |
| Mole % CO ₂ in gas | _ | _ | _ | _ | - | _ | | _ | _ | _ | - | - | - | _ | _ | 0.09 | 0.03 |
| Mole % CO in gas | _ | _ | 0.04 | _ | - | _ | _ | _ | _ | _ | _ | _ | = | | | 0.72 | 0.13 |
| Mole % H ₂ in gas | 97.70 | 99.90 | 99.30 | 98.70 | 97.60 | 98.00 | 98.40 | 97.80 | 98.00 | 98.10 | 98.10 | 98.16 | 97.10 | 95.20 | 97.86 | 98.6 | 97.0 |
| Mole % He in gas | 2.25 | 0.10 | 0.12 | 0.07 | 0.04 | 0.10 | 0.03 | 0.07 | 0.03 | 0.05 | 0.11 | 0.03 | _ | 0.06 | 0.01 | 0.06 | 0.08 |

^a Analysis calculated on water-free basis.

b Starting hydrogen impurities given as follows: less than 5 ppm N2, less than 1 ppm O2, less than 1/2 ppm CO2, less than 1/2 ppm CO, Dew point—100°F.

e Detection limit 0.01%.

d Not analyzed.

Table 5. Mass Spectrographic Analyses of Reaction Gases^{a,b,c} and Wet Chemical Analyses of Residual CO_2 in Unreacted Siderite Siderite-Hydrogen System

| Experiment No. | 86 | 87 | 89 | 40 |
|---|-------|-------|-------|-------|
| Temperature (°C) | 605 | 455 | 400 | 525 |
| Pressure (psi) | 2,000 | 2,000 | 2,000 | 5,000 |
| Duration of run (hours) | 4 | 4 | 4 | 4 |
| Mole % CH4 in gas | 0.15 | 4.34 | 4.45 | 1.45 |
| Mole % C2H6 in gas | | 0.42 | 0.28 | 0.12 |
| Mole % C ₃ H ₈ in gas | | 0.23 | 0.10 | 0.03 |
| Mole % C4H10 in gas | 140 | 0.05 | 0.03 | - |
| Mole % CO2 in gas | | | _ | 0.04 |
| Mole % H2 in gas | 99.80 | 94.90 | 95.10 | 95.30 |
| Mole % He in gas | 0.04 | 0.08 | 0.09 | 3.07 |

^a Analysis calculated on water-free basis.

^c Detection limit 0.01%.

each case to bring the bomb up to temperature before venting and replacing hydrogen for the reaction gas. Because of the low temperature for thermal dissociation of siderite, in those experiments at higher temperatures some siderite undoubtedly decomposed and the decomposition gas, presumably CO₂, was expelled along with the helium. For this reason the mole percent of gaseous hydrocarbons determined in the reaction gas for these experiments are minimum values (Table 5).

The solid reaction products for experiments 40 (525°C, 5000 psi), 86 (605°C, 2000 psi), and 87 (455°C, 2000 psi) are iron (Fe) and wüstite (FeO). The solid reaction produced from experiment 89 (400°C, 2000 psi) is magnetite. In all of the experiments the reaction gases were sampled and replaced with helium at the reaction temperature. Minute and rare flecks of what optically appeared to be graphite were observed, but no certain identification was made.

The limited data available suggests that wüstite is the primary alteration solid. The ultimate $\rm H_2O/H_2$ ratio and the temperature dictate whether wüstite will be reduced to native iron or oxidized to magnetite. The simplified stoichiometric relations would be the following:

$$FeCO_3 + 5H_2 = FeO + CH_4 + 2H_2O$$

if reducing:

$$H_2 + FeO = Fe + H_2O$$

^b Starting hydrogen impurities given as follows: less than 5 ppm N_2 , less than 1 ppm O_2 , less than $\frac{1}{2}$ ppm CO_2 , less than $\frac{1}{2}$ ppm CO_3 , less than $\frac{1}{2}$ ppm CO_4 , Dew point—100°F.