APPENDIX E

HOLD-UP MEASUREMENTS WITH COMPOSITES OF MOBIL REACTOR WAXES

The effect of liquid medium on average gas hold-up was studied using paraffin waxes, reactor waxes and distilled water. The results were presented and discussed in Section V-B.7. One of the reactor waxes used in these studies was a composite of reactor waxes produced from Mobil's runs CT-256-9, -11 and -12. Following these experiments, we planned to further investigate the hydrodynamics of Mobil reactor waxes produced from other runs in Mobil's Unit CT-256 with a two-fold objective. We wanted to better understand the behavior of reactor waxes produced under different conditions and at the same time have results which could be compared with results from Mobil's studies (Kuo et al., 1985) with similar reactor waxes. Two new composites (a composite of reactor waxes produced during runs CT-256-4 and -7, and a composite of reactor waxes produced during runs CT-256-5 and -8) were obtained from the Department of Energy for this purpose.

Experiments were performed in the 0.051 m ID glass column at 265°C and 200°C with the new waxes. The 1 mm and 1.85 mm orifice plates and the 40 μm SMP distributors were employed in these experiments. The effect of small quantities of non-coalescing impurities (1-5% of FT-200 waxes) in the coalescing reactor waxes was also investigated.

The major highlights from these studies are:

- Foam was not produced in any of the runs with the two composites and their behavior was qualitatively similar to that of composite from runs 9, 11 and 12.
- Composite of waxes from runs 4 and 7 showed a slight effect of temperature, with lower hold-ups obtained at 200°C compared to values at 265°C. For all distributors employed, this composite also gave slightly higher hold-ups than either the composite from runs 5 and 8,

or the composite from runs 9, 11 and 12.

- Hold-up values reported by Kuo et al., for similar waxes, are consistently higher than those obtained in the present study.
- When small quantities of FT-200 wax (between 1 and 5% by weight) were added to reactor waxes, hold-ups increased to levels comparable to those obtained in the Mobil study with reactor waxes. A similar increase in hold-up was observed when trace amounts of tap water were added to distilled water.

The effect of superficial gas velocity on hold-ups obtained from experiments conducted with the runs 4 and 7 composite are shown in Figure E-1. These results are qualitatively similar to those obtained using the composite from Mobil's runs 9, 11 and 12 (Figure V-32 in Section V-3.7). Foam was not observed in the runs made with the 1 and 1.85 mm orifice plate distributors. For the run conducted with the 40 μm SMP distributor, foam appeared initially at a gas velocity of 0.01 m/s but disappeared after approximately half hour on stream. The marginally lower gas hold-ups at 200 °C compared to values at 265°C could be attributed to the higher viscosity at 200°C. Results obtained from experiments conducted with the composite from runs 5 and 8 are shown in Figure E-2. Hold-ups from the runs with the different distributors, 1 and 1.85 mm orifice plate and 40 μm SMP, and different temperatures, 200°C and 265°C, are similar. For this composite, unlike with the other two composites, no foam was produced with the SMP distributor at u_g = 0.01 m/s. and hold-ups in the velocity range 0.01-0.04 m/s were lower than values with the other two composites.

Viscosities of Mobil waxes were shown in Table VI-1 (Section VI-A). Measurements made in our laboratory show that the viscosities for the

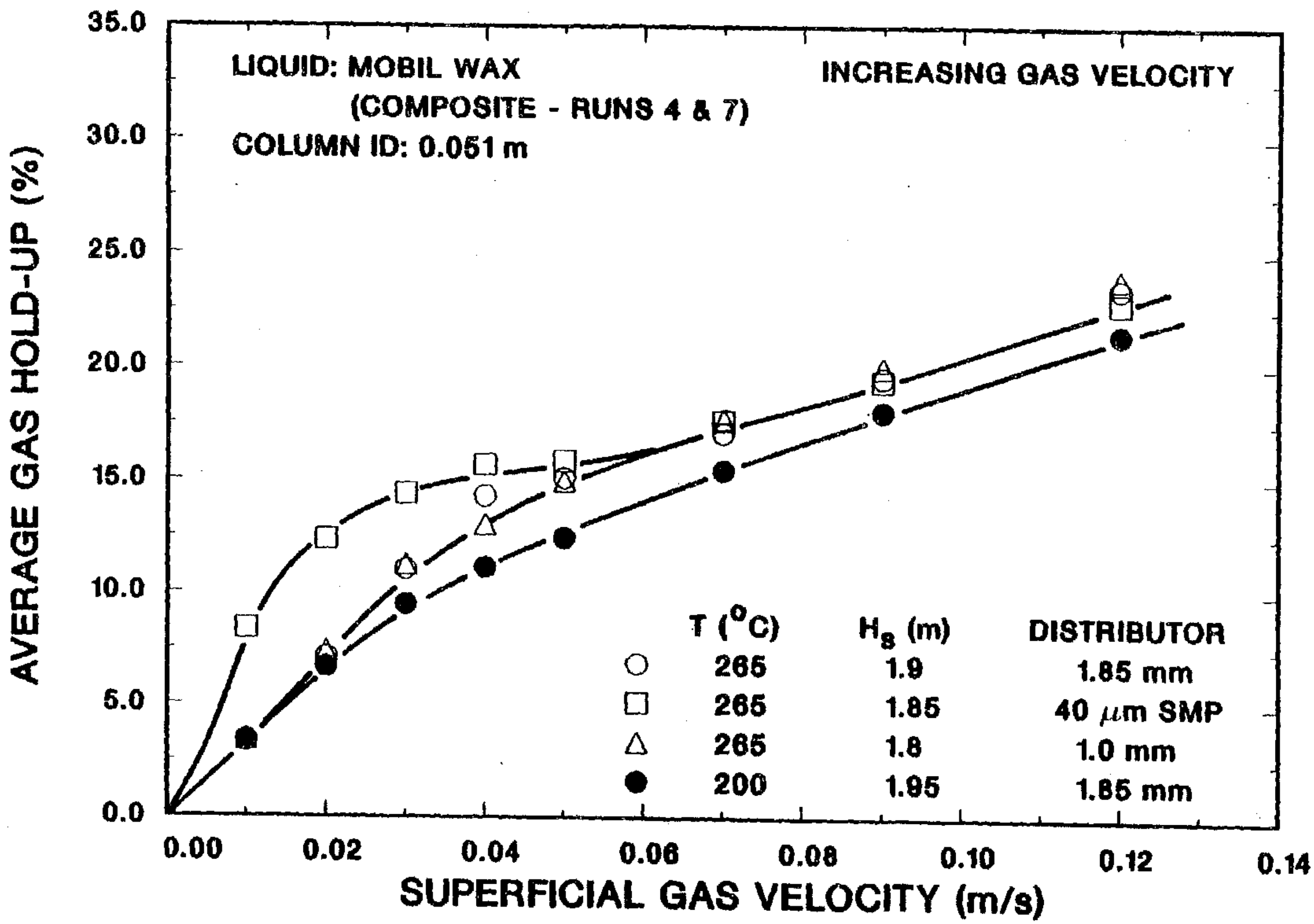

Figure E-1. Effect of superficial gas velocity, distributor type and temperature on gas hold-up (composite wax from Mobil runs CT-256-4 and -7; ○ - Run 21-1; □ - Run 21-2; △ - Run 21-3; ● - Run 21-4)

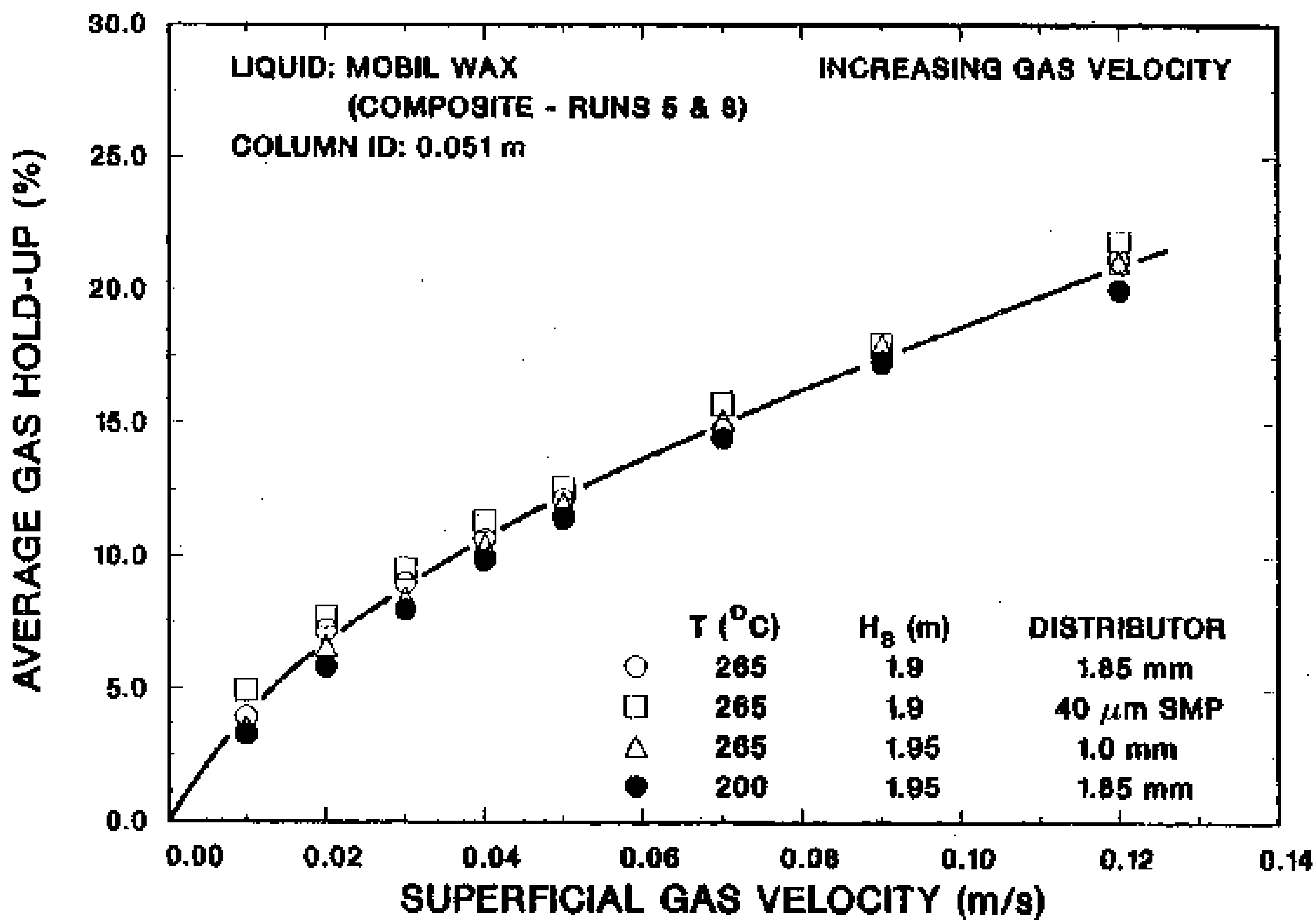

Figure E-2. Effect of superficial gas velocity, distributor type and temperature on gas hold-up (composite wax from Mobil runs CT-256-5 and -8; ○ - Run 22-3; □ - Run 22-1; △ - Run 22-2; ● - Run 22-4) composite from runs 4 and 7 were lower than those for the composite from runs 5 and 8 in the temperature range used during these measurements (T ≤ 230°C). However, when viscosity of the composites were estimated at 265°C from data at lower temperatures, assuming Arrhenius type of dependency of viscosity with temperature, similar values were obtained for the two composites. This leads us to believe that the difference in behavior between the two composites, with the SMF distributor, is probably due to differences in the compositions of the two waxes.

Figure E-3 compares hold-up values obtained with the three Mobil wax composites when experiments were conducted at 265°C using the 1.85 mm orifice plate distributor. Earlier comparison of hold-ups obtained when different liquid media (including the composite from runs 9, 11 and 12) were used (Figure V-34, Section V-B.7) showed that, in the "slug-flow" regime, hold-ups with the different liquids were very similar. Results shown in Figure E-3 further support these findings. Hold-up values with the composite from runs 4 and 7 are consistently higher then values for the other two composites, however, the difference is not significant (between 2% - 3% absolute).

Hold-up values obtained by Kuo et al. (1985) from experiments conducted at 260°C with the run 7 wax and the run 8 wax using a 1 mm orifice plate distributor, are shown in Figure E-4. Also shown are hold-ups obtained from the present study, with the composite from runs 4 and 7, and the composite from runs 5 and 8, under similar conditions. Reactor waxes produced in runs 4 and 7 have similar physical properties (Table VI-1; Section VI-A) and their compositions are expected to be similar. The same is true for reactor waxes produced in runs 5 and 8. However, results

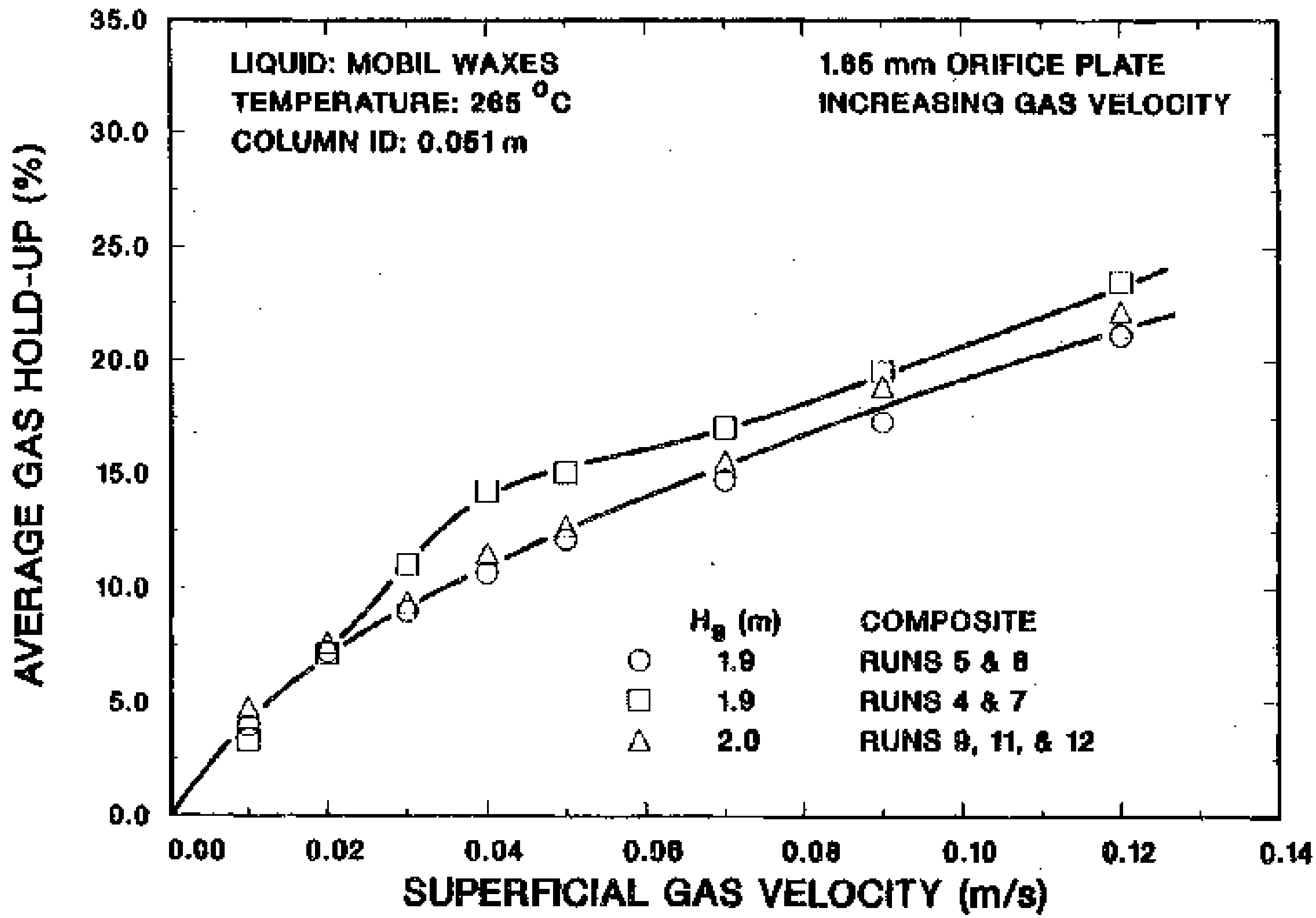

Figure E-3. Comparison of hold-ups obtained in studies with different Mobil wax composites (○ - Run 22-3; □ - Run 21-1; △ - Run 9-3)

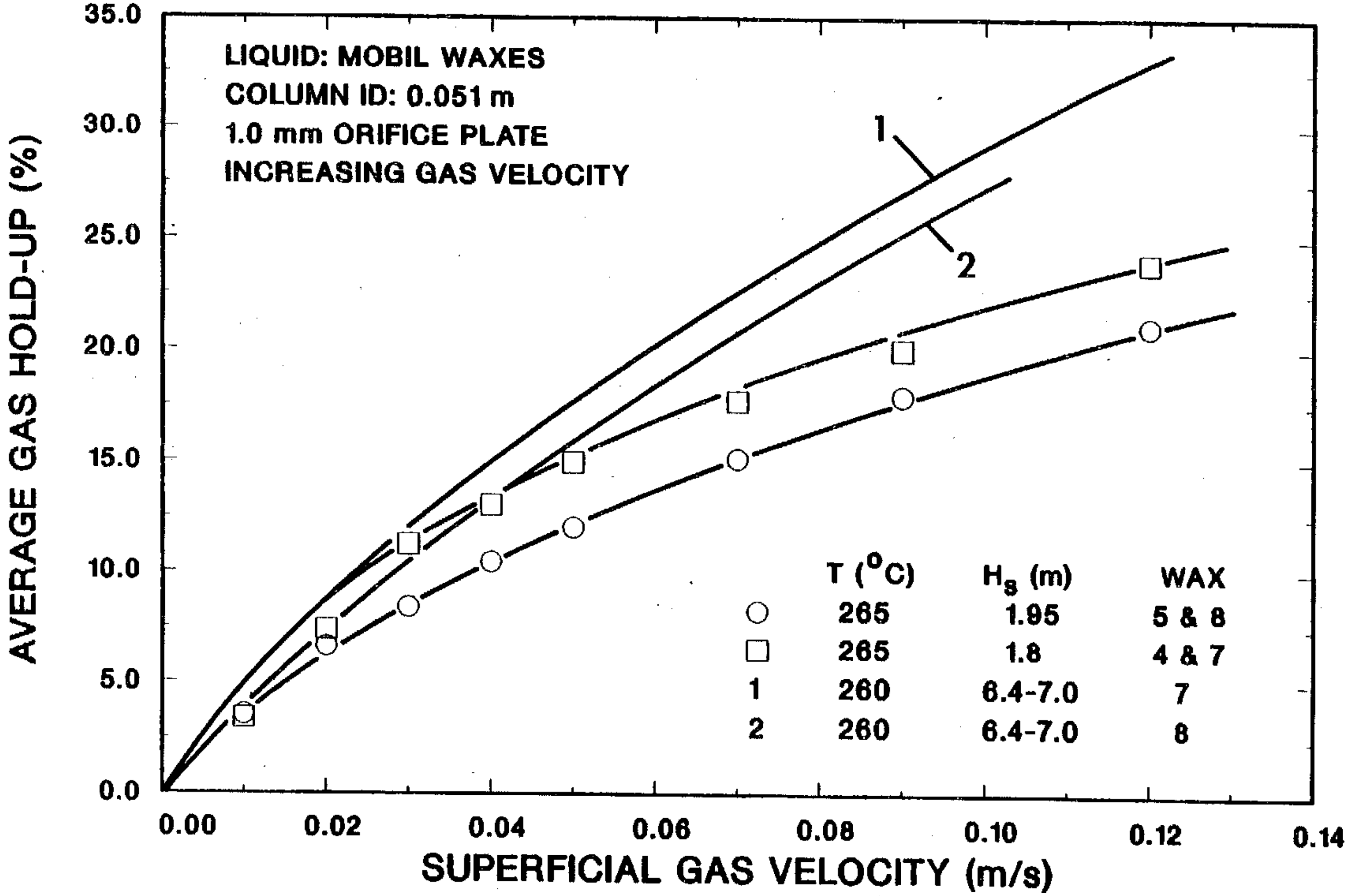

Figure E-4. Comparison of hold-ups obtained in present study with literature values (1,2 - Kuo et al., 1985; Symbols - present study; Wax type is indicated by run numbers in Mobil's Unit CT-256 during which wax was produced; ○ - Run 22-2; □ - Run 21-3) presented in figure E-4 show that hold-ups obtained by Kuo et al. with the run 7 wax are significantly higher than those obtained by us with the composite from runs 4 and 7 for $u_g > 0.03$ m/s. Similarly, results obtained by them with the run 8 wax are higher than those obtained in the present study with the composite from runs 5 and 6 for $u_g > 0.02$ m/s. The only difference in the conditions employed in the two studies (Kuo et al. and the present study) is the difference in static heights. Kuo et al. used static heights in the range 6.4 - 7.0 m, whereas static heights in the range 1.8 - 2.0 m were used in our experiments. For coalescing media, such as reactor waxes, we do not expect taller beds to have higher hold-ups. This is because the small bubbles near the distributor would coalesce into fast rising large bubbles or slugs. These would lower the hold-up in the top-half of the column; implying lower hold-ups for taller columns. Evidence to support this is available from axial gas hold-up profiles obtained by Kuo et al. for experiments conducted using the run 7 reactor wax in the 0.051 m ID by 9.1 m tall column (Kuo et al., 1985). Their results show that for $u_g > 0.025$ m/s, gas hold-up decreased with height. This suggests that hold-ups in a taller column should be lower than those obtained in a shorter column. Thus the differences in static heights cannot be used to explain the discrepancies in hold-up values from the two studies.

The experiments by Mobil, with reactor waxes in tall columns, were done after conducting experiments with FT-200 wax (foaming system). Thus, small amounts of FT-200 left on the column wall might have contaminated the reactor waxes and affected the gas hold-up. This effect was investigated in our laboratory by adding small quantities (between 1% and 5%) of FT-200 to reactor waxes, and measuring hold-ups. Hold-ups, with a mixture of 3% by weight FT-200 and composite from runs 4 and 7, were significantly higher than hold-ups for the pure composite (Figure E-5), and were comparable with values reported by Kuo et al. for the run 7 wax. Similarly, when 5% by weight of FT-200 was added to the composite from runs 5 and 8, hold-ups were higher than those observed with the pure composite (Figure E-6), though somewhat lower than those reported by Kuo et al. for $u_g > 0.05$ m/s. These results show that small amounts of non-coalescing impurities present in a coalescing medium, can lead to a significant increase in gas hold-ups. Similar behavior was observed for runs conducted with distilled water. When experiments were done with distilled water, following runs with tap water in the same column, higher hold-up values were obtained compared to values from a run in a clean column (Figure E-7). This was caused by the small amounts of tap water remaining on the column wall. Hold-ups for the contaminated distilled water are between those for distilled water and tap water (Figure E-7).

The experiments with reactor waxes having non-coalescing impurities, and with contaminated distilled water show that, in order to get true gas hold-ups for coalescing media, special care needs to be taken to prevent non-coalescing impurities from entering into the system. Only small amounts of such impurities can lead to significantly higher hold-ups for the coalescing media.

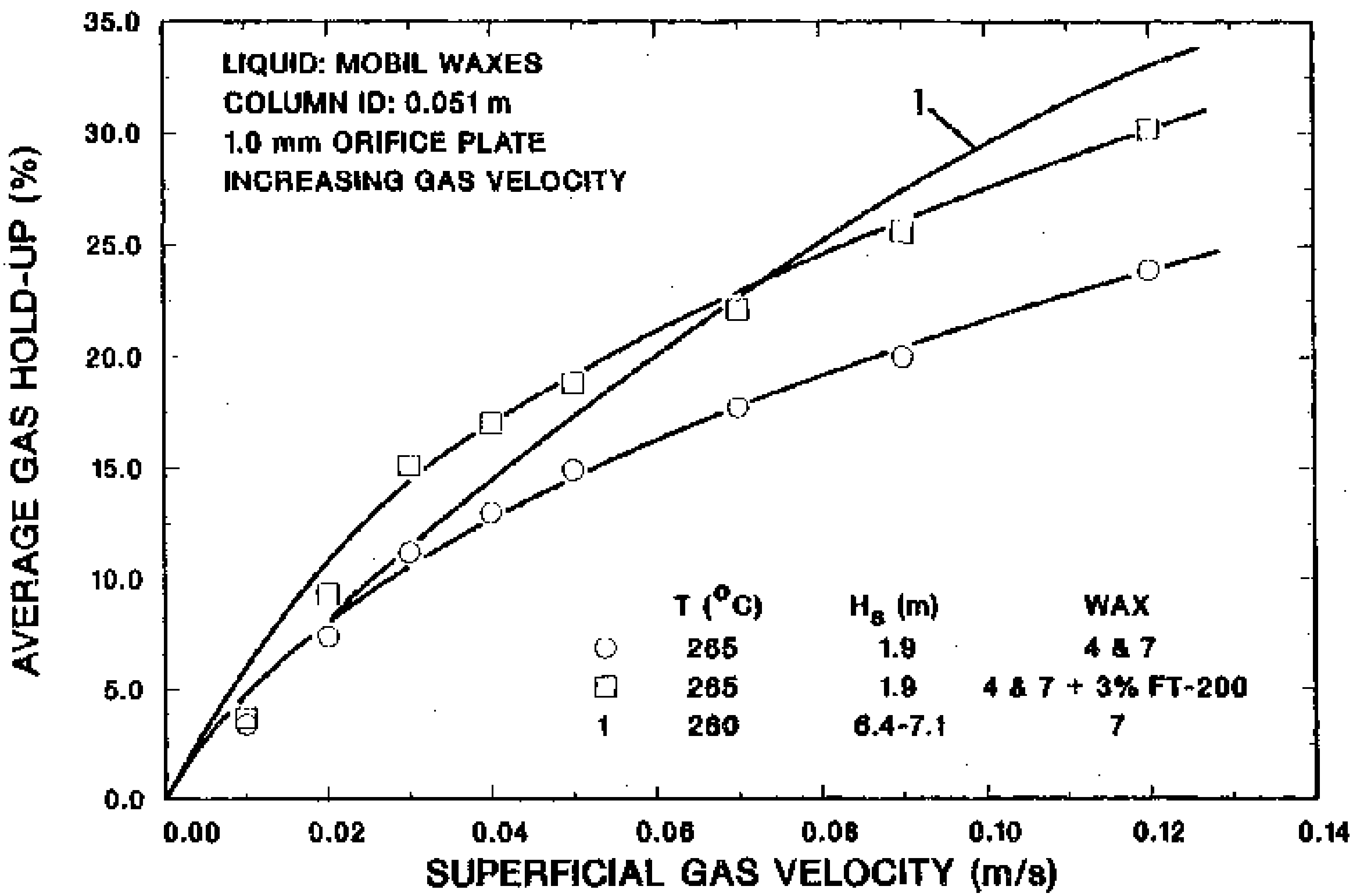

Figure E-5. Effect of small quantities of FT-200 on hold-up (1 - Kuo et al., 1985; Symbols - present study; Wax type is indicated by run numbers in Mobil's Unit CT-256 during which wax was produced; ○ - Run 21-3; □ - Run 23-3)

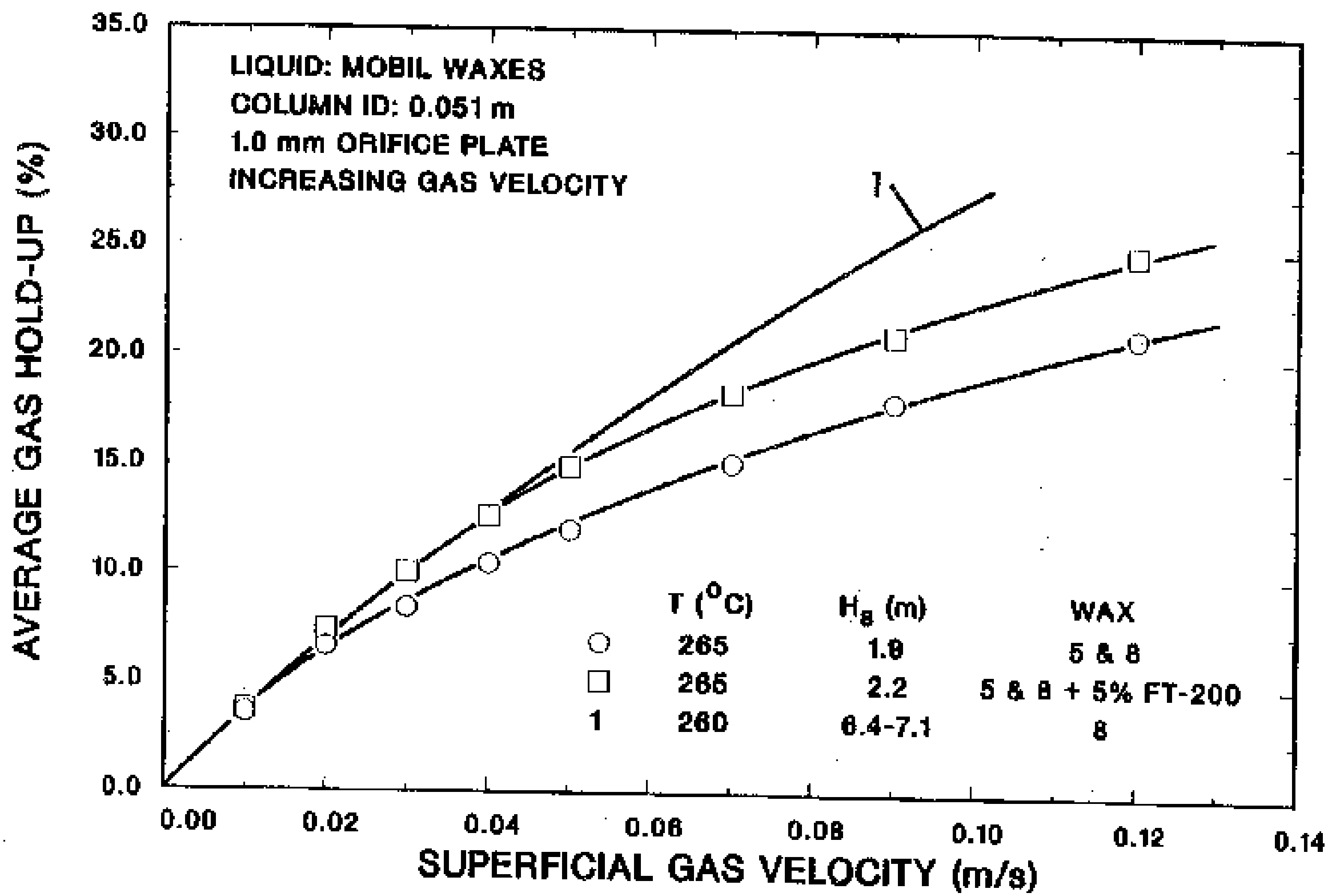

Figure E-6 Effect of small quantities of FT-200 on hold-up (1 - Kuo et al., 1985; Symbols - present study; Wax type is indicated by run numbers in Mobil's Unit CT-256 during which wax was produced: ○ - Run 22-2; □ - Run 24-3)

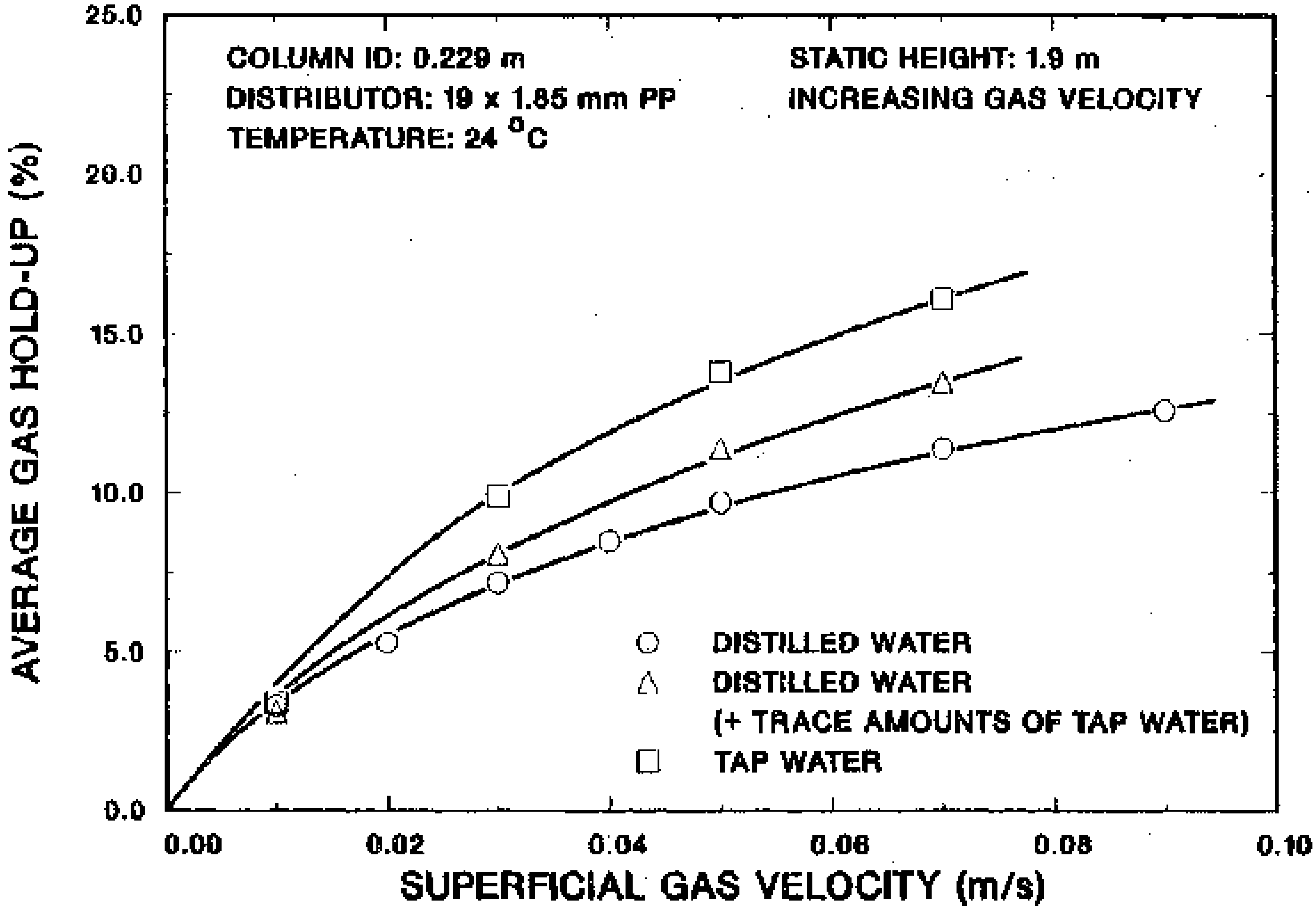

Figure E-7. Effect of contamination on hold-ups with distilled water