Recirculation Systems

Figure 4 shows schematically the gas and liquid recirculation systems.

All connecting lines are $^1/_8$-inch stainless steel tubing, except that two sections carrying the gas phase are $^1/_4$ inch; the latter are represented by a heavier line.

The mixing and equilibration of the phase take place in the equilibrium cell, 10. The gas phase flows from the top of the cell through the modified safety head, 11, and through the three-port valve, 12, which is capped with cap 13. From valve 12 the gas flows through a 300-cc. gas-surge vessel, 14, and then to either linear valve 15 or block valve 16. When the plunger of linear valve 15 is pulled out, the recirculating gas passes through the sample loop and then to the small gas-surge vessel, 17. When the plunger is pushed in, the sample loop is connected to the carrier gas lines of the gas chromatograph and the sample is thus injected for analysis. When the plunger is in, block valve 16 is opened to allow the recirculating gas to bypass linear valve 15. Gas in the small gas-surge vessel, 17, is pumped by magnetic pump 18 through three-port valve 19, which is used to admit gas to the system. Gas from valve 19 passes through a tee connection which is connected to capped (cap 9) transducer receiver 20, into which is screwed the flush-diaphragm pressure transducer, 41. From the tee connection the gas flows through valve 21 and back to the equilibrium cell, 10, where it enters just below the upper liquid level.

In the original design of the apparatus the recirculating gas flowed through the transducer receiver and across the face of the pressure transducer diaphragm, so that all dead space was eliminated—i.e., no tee connection was used. A change of the gas flow to go through transducer receiver 20—i.e., by placing transducer receiver 20 in the $^1/_4$-inch line between valve 19 and valve 21—is still desirable for the further elimination of dead space.

After the plunger of linear valve 15 has been pushed in, the gas sample loop will normally be filled with carrier gas. The compressed gas being studied is used to purge the loop. The same gas is then used to pressure the loop to a pressure somewhat above that in the equilibrium apparatus and the plunger is pulled out to begin sampling.

The heavy liquid is withdrawn from the bottom of equilibrium cell 10 by magnetic pump 22, from which it flows through valve 23, liquid sampler 24, three-port valve 25, and three-port valve 27. From valve 27 the liquid returns to equilibrium cell 10 and is directed away from the wall of the cell by a welded ramp.

The recirculation system for the less dense liquid is identical to that for the denser liquid, except that the less dense liquid is withdrawn from the equilibrium cell through a port underneath the heavy-liquid inlet ramp and is later returned below the level of the heavy liquid. Parts 32 to 37 of the light-liquid recirculation system correspond in function to components 22 to 27 of the heavy-liquid system.

During equilibration, the recirculating heavy liquid flows through liquid sampler 24 and bathes the hole in the end of the threaded shaft. When the sample is to be trapped, the threaded shaft is screwed out the proper number of turns to trap the sample. Valves 23 and 25 are closed and the sampler is removed and purged with carrier gas. On injection the threaded shaft is screwed in to release the trapped sample into the carrier gas for analysis.

After analysis, sampler 24 is fitted to valves 23 and 25, cap 26 is loosened, and valve 23 is opened slowly to allow a small amount of liquid to flash into the sampler to purge out the air. Cap 26 is then tightened, valve 23 is opened, and then valve 25 is opened.

A large number of operating details are necessary for the successful startup, maintenance, and shutdown of the apparatus. These and other details of the apparatus are reported elsewhere (Fleck, 1967).

Experimental Data

Figure 5 shows gas-phase data obtained for the binary system ethylene–*n*-propyl alcohol at 15° C. With this binary system both liquid-gas and liquid-liquid-gas equilibria were encountered. A minimum solubility of *n*-propyl alcohol in the gas phase was found at about 410 p.s.i.a. Figure 5 shows the relatively good reproducibility of the data at very low concentrations of *n*-propyl alcohol.

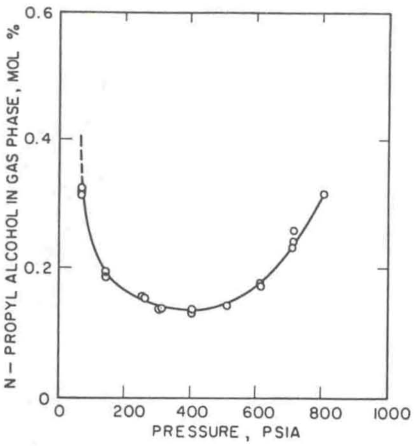

Figure 5. Gas-phase data

System. Ethylene–*n*-propyl alcohol at 15°C.

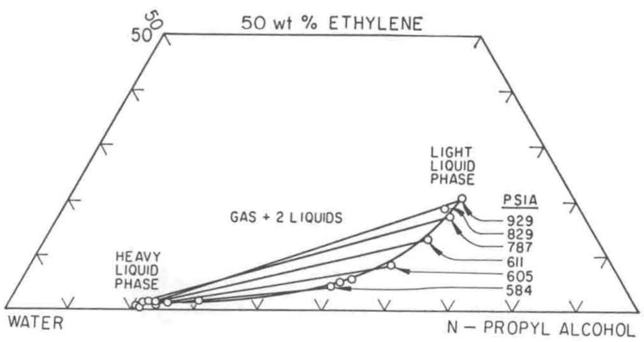

Figure 6. Liquid-liquid tie lines

System. Water–*n*-propyl alcohol–ethylene at 15° C.

Figure 6 shows the liquid-liquid tie lines for several pressures at 15° C. in the liquid-liquid-gas region of the ternary system water–*n*-propyl alcohol–ethylene. Interpolated data for 715 p.s.i.a. agree reasonably well with those obtained by Elgin and Weinstock (1959).

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Literature Cited

Elgin, J. C., Weinstock, J. J., *J. Chem. Eng. Data* **4**, 3 (1959).

Fleck, R. N., dissertation, Department of Chemical Engineering, University of California, Berkeley, 1967.

Stein, F. P., Sterner, C. J., Geist, J. M., *Chem. Eng. Progr.* **58** (11), 70 (1962).

Sterner, C. J., *Rev. Sci. Instr.* **31**, 1159 (1960).

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