Notes from the Oesper Collections

The Craig Countercurrent Distribution Train

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The Oesper Apparatus Museum has recently had the good fortune to acquire a 30-cell Craig countercurrent distribution train. First described in 1949 (1) by the American biochemist, Dr. Lyman C. Craig (figure 1), of the Rockefeller Institute for Medical Research, and, in a more refined form, in 1951 (2), the Craig apparatus formed a key advance in liquid-liquid extraction techniques which bridged the era of the classical separatory funnel, on the one hand, and that of the wide-spread adoption of liquid chromatographic techniques, on the other. The unit was donated by Dr. Robley Williams, a former student of Craig, who also drove it across country from his home in Colorado to deposit in the Oesper Museum. To the best of Dr. Williams' knowledge it is the only surviving example of Craig's invention outside of the unit currently on display in the lobby of the Rockefeller Institute in New York City.

According to Schindler, the separatory funnel (figure 2) did not attain its current form until the 1850s (3).

Figure 2. A traditional separatory funnel, c. 1880. (Jensen-Thomas Apparatus Collection).

Figure 1. Lyman C. Craig (1906-1974).

Its use to separate the components of a mixture is complicated by the fact that the distribution of a solute between two immiscible liquid phases is an equilibrium process governed by the famous Nernst distribution law. This means that the extraction of the desired product is never complete and that the undesirable components of the mixture are often partially extracted as well. The only way to improve the situation is to repeatedly add fresh aliquots of the extracting solvent and repeat the separation – a process which, if performed manually using the traditional separatory funnel, can be extremely tedious if the degree of separation per extraction step is low.

What Craig did was to essentially automate this process by replacing the separatory funnel with a specially designed equilibration cell (figure 3). The cell, which contained the extracting solvent, was originally placed in position A and the solution containing the sample mixture added though b. The cell was then

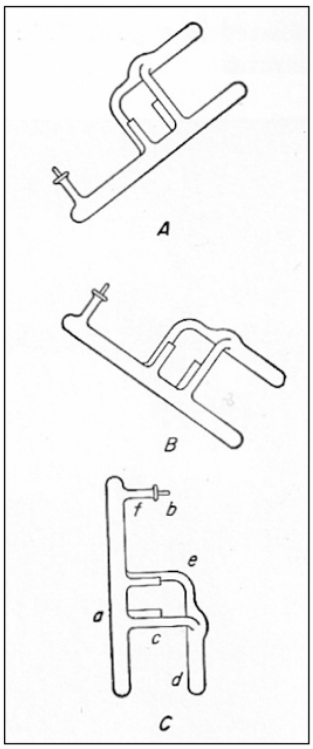

Figure 3. The operation of an individual equilibration cell.

rocked back and forth between positions A and B to equilibrate the two phases. Stopping the rocking motion in position B until the two liquid phases had once again separated, the cell was then rotated to position C, which allowed the lighter sample solution to decant through tube c into chamber d. The cell was then rotated back to position A, which allowed the sample solution to exit the cell through tube e, which was connected to the adjacent cell containing a fresh increment of the extracting solvent, and the entire process repeated. Anywhere from 25 to over 200 cells could be linked together depending on the difficulty of the separation. In the smaller units the cells were rocked by hand around a common pivot using a crank. In the case of the large 220-cell unit used by Craig himself (figure 4), which occupied an entire bench top, it was rocked using a motor and a timer. Depending on the number of cells in the train, it could take up to 24 hours for the mixture to pass through the complete series of extractions.

The model donated by Dr. Williams (figure 5) contains 30 cells arranged in two rows and is operated by hand. It was manufactured by the H. O. Post Scientific Instrument Co. of Middle Village, New York (figure 6). Otto Post was the machinist and glassblower at the Rockefeller Institute and had built the various prototypes of Craig's device. German born, he had served

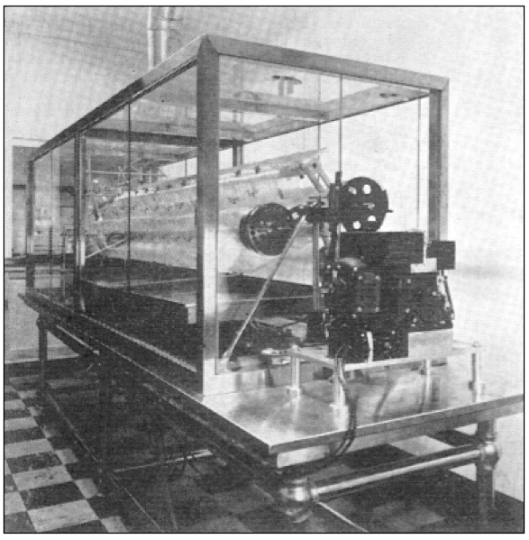

Figure 4. Craig's 220-cell countercurrent extraction train at the Rockefeller Institute. circa 1951 (2).

as a sharpshooter instructor in the German army during World War I.

After the extraction was complete, one still had to isolate the separated final product by removing the extracting solvent. If, as was frequently the case with biochemical substances, the desired product was thermally sensitive, this precluded strong heating to induce rapid evaporation. If the product was air sensitive, this precluded the much slower process of allowing the solvent to gradually evaporate in the open air at room temperature. Attempts to resolve this problem led to

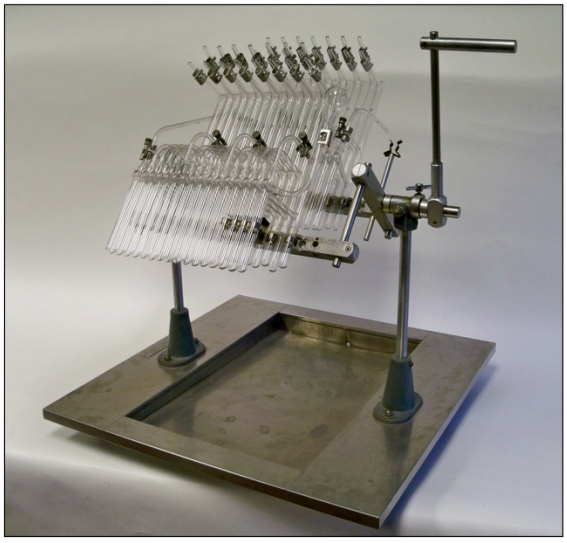

Figure 5. The double-row, 30 unit Craig countercurrent separation train donated by Dr. Robley Williams (Jensen-Thomas Apparatus Collection).

Figure 6. The instrument-maker's label on the Craig distribution train donated by Dr. Williams.

Craig's second important innovation in chemical apparatus – the well known laboratory rotavap – which he first proposed in 1950 (4) and of which the Oesper collections owns at least six early models (figure 7). While the gradual displacement of the Craig countercurrent separation train by ever more powerful and convenient chromatographic techniques was perhaps inevitable, the rotavap continues to retain its status as an indispensable part of both the organic and biochemical laboratory.

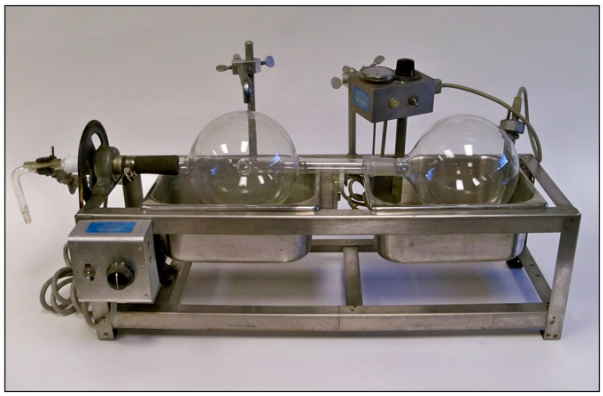

Figure 7. A Buchler Flash-Evaporator – an early American commercial version of Craig's rotavap produced by the Buchler Instrument Company of New Jersey, c.1964 (Jensen-Thomas Apparatus Collection).

References and Notes

1. L. C. Craig, O. Post, "Apparatus for Countercurrent Distribution," *Anal. Chem.*, **1949**, *21*, 500-504.

2. L. C. Craig, W. Hausmann, E. H. Ahrens, E. J. Harfenist, "Automatic Countercurrent Distribution Equipment," *Anal. Chem.*, **1951**, *23*, 1236-1244.

3. H. Schindler, "Notes on the History of the Separatory Funnel," *J. Chem. Educ.*, **1957**, *34*, 528-530.

4. W. B. Jensen, "Origins of the Rotavap," *J. Chem. Educ.*, **2008**, *85*, 1481.