

# AN INSTRUMENT FOR THE DEMONSTRATION OF COUNTERCURRENT CONTINUOUS PROCESSES

By T. SZÉLL

Institute of Applied Chemistry, The University, Szeged  
and

L. SAMU

Glass-Blowing Shop of the University of Szeged

(Received January 15, 1961)

An instrument made of glass has been described for the demonstration and study of counter-current continuous processes.

## § 1. Introduction

The importance of countercurrent continuous reactions and processes is well known. The simple device, shown below, is one of those apparatuses which are suitable for the demonstration of the principle of countercurrent and continuity, as well as for the laboratory exercises of students in chemistry. Thus, the instrument may be used in high schools and universities, both for demonstration and experimenting in connection with chemical engineering operations as well as with the instruction of chemical technology.

## § 2. Description of the apparatus

The apparatus consists of 5—10 reactor-units, made of glass (Fig. 1). Fig. 2 shows a set-up of five units. The liquid of a relatively low density is fed from the separating funnel, placed on a higher level, and the liquid passes in conformity with the principle of the communicating vessels, through the heat exchanger coil (which may be omitted in certain given cases) and the reactor-units. The material of higher density, fed from the other separating funnel, runs by gravitation. The reactors may also be made in such a manner (Fig. 3), as to permit to fill it with glass beads or RASCHIG rings, in order to make the mixing of the two phases still more complete.

## § 3. Some examples of the practicability of the apparatus

a) With the apparatus, e. g., the countercurrent continuous extraction may be demonstrated. Obviously, a great number of pairs of materials is suitable for the demonstration of the liquid-liquid extraction. Thus, may conveniently be used,

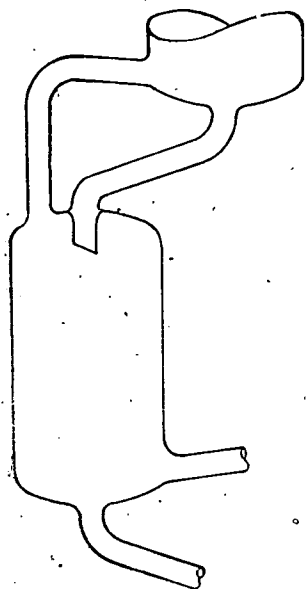


Fig. 1

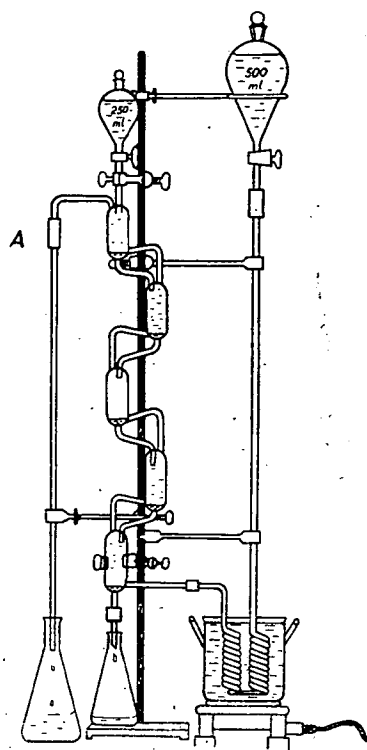


Fig. 2

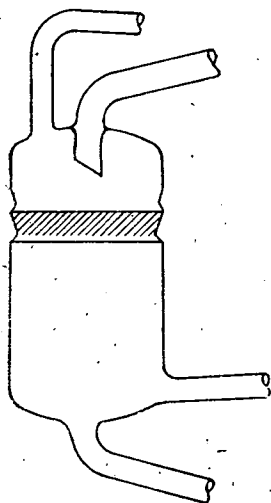


Fig. 3

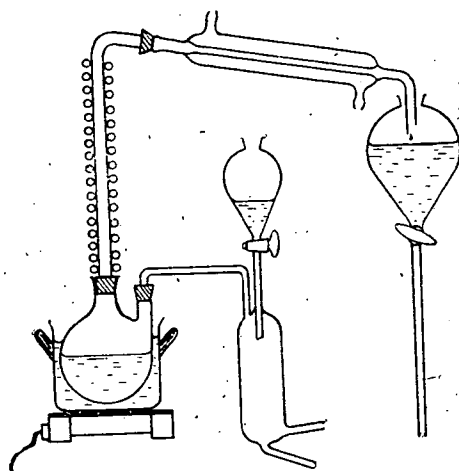


Fig. 4

e. g., picric acid, dissolved in benzene (1 g per litre) together with 1 N aqueous solution of NaOH. The fading of the hue of the yellow benzene solution and the turning yellow of the caustic — at the beginning colourless — solution (sodium picrate), may be easily followed with naked eyes or by means of colorimetry. An example of industrial character is the extraction of phenols from gasworks liquor, previously neutralized and coloured violet with ferric chloride, by means of esters (ethyl- or butyl-acetate). Here, the gradual discolouration of the heavier aqueous part may be observed in consequence of the dissolution of phenols.

b) The apparatus may be used for carrying out countercurrent *reaction*, too. Thus, e. g., sulfuric acid refining of crude benzene and tar or petroleum products, as well as other heterogeneous liquid-liquid reactions may be carried out with this device.

As a typical example, we shall further on expose the continuous sulfonation of benzene, which will demonstrate at the same time the operation of the apparatus as well.

#### § 4. *Manufacture of benzene sulfonic acid*

This process is a well known industrial procedure in its *continuous* form, too [1], although benzene sulfonic acid may also be produced by a batch type reaction without any difficulty [2].

The benzene gets into the apparatus through a spiral, plunged into a water bath of 75° C, while at the same time a 12—20 percent oleum is fed from the smaller funnel at such a rate as to ensure the formation of a 8—12 mm thick permanent seal of oleum in the bottom reactor. It is, of course, important that the outflow of the oleum at the bottom be of the same rate as its feeding above. At equal length of time about three times as much ml of benzene should pass through the apparatus as oleum. The oleum may repeatedly be used as long as its concentration does not drop to 2 or 3 per cent. The oleum dissolves the sulfonic acid better than benzene does, therefore, it is more expedient — for the sake of realizing better conditions of distribution — to use such oleum which already has a content of sulfonic acid, *i. e.*, which had already passed through the apparatus. The ageing of oleum may be controlled with the help of a densitometer. The benzene solution, containing some tenth part of per cent of benzene-sulfonic acid is made flown — advantageously, at a rapid pace of dropping, as shown by Fig. 4 — into the flask which is joined at spot „A” to the apparatus, shown by Fig. 2. The solvent is then removed from the flask by means of electric heating (open flame would mean a fire hazard!), at such a rate as it is required. As it is also shown in Fig. 4, the benzene obtained in the course of distillation takes part again in the process, while the sulfonic acid becomes concentrated in the flask. In order to obtain the product, one may either follow the semi-continuous procedure described below or else carefully evaporate the benzene and make the residue flow into an ice-cold saturated NaCl solution, when — after allowing to stand for about 2 hours in a refrigerator — the  $\text{PhSO}_2\text{ONa}$  crystallizes in the form of small platelets [2].

*Semi-continuous procedure:* If, for any reason, the modification sketched by Fig. 4 is not at disposal, then benzenesulfonic acid may be produced in the original setting, in a more primitive way, too. In this case the benzene flowing through (400 ml) is made pass six or seven times again through the apparatus, while the

oleum (200 ml) is replenished about four times. The benzene, flowing out at the end of the test is then washed with  $4 \times 20$  ml of water, 3 g NaCl are added to the combined aqueous extract, and following the dissolution it is concentrated to a volume of 10 ml. The solution thus obtained, practically saturated regarding NaCl is placed in a refrigerator, where  $\text{PhSO}_2\text{ONa}$  crystallizes in about two hours. The benzene washed out, may be used again, after drying ( $\text{Na}_2\text{SO}_4$ ).

From time to time, a part of the spent acid must be taken away, while stronger oleum (e. g. a 20 per cent) shall be added to the spent sulfonating acid in the required proportion, the thus obtained oleum will be again utilized. The oleum taken away, is flown into a saturated ice cool NaCl solution of about a 3,5-fold volume. This way, from each 10 ml part of oleum a further yield of 2—6 g of  $\text{PhSO}_2\text{ONa}$  may be obtained in a way identical with the above described one.

In the first experiment, the production is somewhat smaller, since at the beginning the apparatus is filled with pure benzene, while in the consecutive tests, the benzene in the apparatus already contains sulfonated products, too.

\* \* \*

The authors wish to express their gratitude to Á. FURKA for his helpful suggestions.

#### References

- [1] *Groggins, P. H.*: Unit Processes in Organic Synthesis (McGraw—Hill Publ. Co. Ltd., London, 1947), p. 321;  
Monsanto and Dennis-Bull processes, see *K. Winnacker, Weingaertner, E.*: Chemische Technologie; Organische Technologie II (C. Hanser Verl., München, 1954), p. 18;  
*Barbet*: U. S. P., 1.459.081 (1923).  
*Ambler and Gibbs*: U. S. P. 1.300.228 (1919), Color Laboratory.
- [2] *Gattermann, L., H. Wieland*: Die Praxis der organischen Chemikers (W. de Gruyter and Co., Berlin, 1948), p. 175;  
*Groggins, P. H.*: Unit Processes in Organic Synthesis (McGraw-Hill Publ. Co. Ltd., London, 1947), p. 320.

#### СРЕДСТВО ДЛЯ ИЛЮСТРАЦИИ БЕЗПРЕРЫВНЫХ ПРОЦЕССОВ ПРОТИВНОГО ТЕЧЕНИЯ

Т. Селл и Л. Шаму

Для изучения и иллюстрации процессов непрерывного противного течения было написано одно стеклянное средство.