

The Szombathy-Soxhlet Extractor

In discussing the unfavorable equilibrium constant in which only a few percent of aldol product is formed from acetone (p. 433) our text notes, “However, if the product is removed from the basic catalyst as it is formed, the conversion can be accomplished in 80% yield.”

At first glance this may sound trivial, but deciding how to do it is a challenge. One might imagine separation by filtration or distillation. In fact both are required. If a solid, insoluble base is used (BaO), it can be removed from the fluid by filtration. The dominant constituent of the fluid, ~96% acetone, can be removed by distillation, but only after the base is removed, so that the dimer will not revert to monomer as the monomer distills away.

So one can imagine multiple repetition of the following sequence, each cycle converting about 4% of the acetone to its aldol dimer and recycling 96% :

- 1) Let acetone stand with powdered BaO for several minutes to approach an equilibrium concentration of the dimer (note that time and surface area are necessary for effective equilibration catalyzed by an insoluble base).
- 2) Filter to remove BaO.
- 3) Remove acetone by distillation.
- 4) Add acetone to BaO and return to 1.

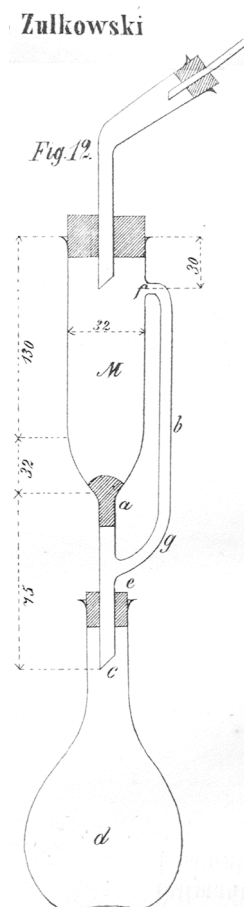
After repeating this procedure 17 times one would have converted about half of the initial acetone to dimer, **IF** there were no losses in the filtration and distillation. There are always losses. Losing 4% of the acetone on each cycle would cut the ultimate yield of aldol product in half.

There must be a better way.

One way would be to use the automatic extraction apparatus invented by Professor Zulkowski from the Technical Institute in the Czech city of Brno in 1873. ⁽¹⁾ The solid catalyst could be held in the “funnel,” *M*, by a plug of cotton, *a*, while acetone is repeatedly distilled from flask *d* through sidearm *b* to be condensed at the top and drip back through the catalyst. Everything would be automatic, and there would be no loss of acetone from handling (assuming good corks). Ultimately the acetone would all be consumed and flask *d* would contain only aldol. The only inefficiency might be from the acetone’s not staying long enough with the BaO to approach equilibrium. (There is also the problem that given enough time the aldol might dehydrate to α,β -unsaturated ketone.)

An even better choice would be to use a Soxhlet extractor. Franz von Soxhlet (1849-1926), a native of Brno, was a chemist at the Vienna Agricultural Institute with a particular interest in milk. He was the first to describe milk sugar (lactose) and was referred to as the “reformer of infant nutrition” because of his work developing individual sterilized portions of baby formula.

In 1879 Soxhlet proposed a new way to measure the amount of fat in milk. ⁽²⁾ He would remove the water from 10 mL of milk by absorbing it with 20 g of dry CaSO₄, wash the fat from the resulting powder with ether, evaporate the ether, and weigh the fat. He thought of using Zulkowski’s apparatus to wash the powder with the same portion of



ether repeatedly, but he found that it took 10 hours to make the washing complete. True, the device was automatic, but 10 hours was a long time.

Soxhlet found the better way, “The following procedure allows the result to be achieved in less than two hours, realizing this important acceleration without in the least compromising either accuracy or certainty...

He saw that the problem was that the powder was not efficiently exposed to the ether, which also did not sit long enough with the powder to become saturated. Moreover the ether exposed to powder was constantly being diluted by freshly distilled ether.

As Soxhlet wrote: “Thus for example complete extraction of a substance using the widespread Zulkowsky apparatus (*this journal*, **1873**, 208, 298) usually takes 10 hours. The long duration of the washing process required by this or a similar apparatus comes from the fact that, like all other automatic extraction devices that have recently been recommended, it works according to the particularly frowned upon principle of continuous washing. Ether drips from the reflux condenser onto the substance, the fat-containing ether drips down, the new ether dilutes the fat solution without immersing the substance. In short the operation of this apparatus is subject to all the defects inherent in continuous washing methods, and because of which it is excluded from the catalogue of analytical operations. The automatic extraction apparatus should work in the same way as a precipitate is normally washed by filtration, where it is immersed in the washing liquid, which is renewed only after the previous liquid is poured or drained off.”

Fortunately Soxhlet’s clever Hungarian student devised a solution: “The problem of transforming a continuous fat extraction apparatus into one that automatically drains the substance as in normal washing, has been solved in this laboratory by Mr. *Szombathy* in a very ingenious and simple manner by using a lifting device that sucks away the ether solution of fat after it has reached a certain height in the extraction vessel. After several preliminary experiments that I conducted, the most convenient dimensions of its various parts, and the conditions for its proper operation, were discovered. The construction and mode of operation of the apparatus is easily understood from the following sketch.”

Liquid siphons completely into the flask attached at the bottom of the Szombathy-Soxhlet device only after it has filled vessel *A* to the height *h*, so as to prime the siphon, *D*. Soxhlet found that a cylinder of filter paper crimped at the bottom was adequate to prevent powders from siphoning off with the ether. Note that all of the saturated ether is removed before *A* begins refilling with freshly redistilled ether.

Certainly Soxhlet deserves significant credit for the extractor that is still used, but it is a pity that Mr. Szombathy has been lost to history.

References:

<http://www.whonamedit.com/doctor.cfm/3041.html>

- (1) “On a New Extraction Apparatus,” Zulkowski, *Dingler’s Polytechnisches Journal*, **1873**, 298-300.
- (2) “Determination of Milk Fat by Weight”, F. Soxhlet, *ibid.*, **1879**, 32, 461-465.

