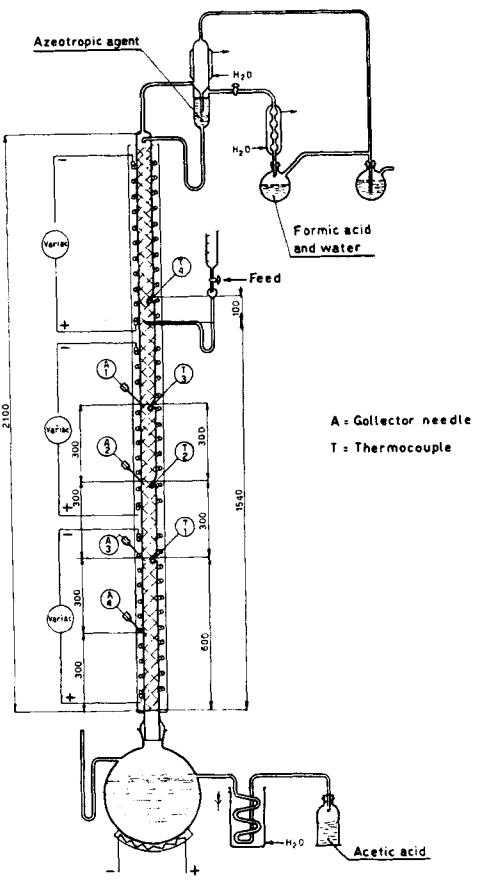
TECHNICAL NOTE

Measurement of the composition of a system along a distillation column by labelled compounds *

The calculation of the number of theoretical plates, in a distillation column, may become very difficult if applied to systems largely differing from ideality or to more complicated mixtures requiring a very tedious treatment of the available data.



* Received on 26 July 1965.

In these cases, it is possible to collect the data for the calculation of an industrial column by means of a small laboratory column, packed with Fenske rings. The composition of the system along the column, besides that of the distillate and of the residue, must be known precisely for a complete evaluation of the separation. It is however difficult to take off, from a small column, the amounts of product needed for analyses, without changing the operative conditions of the column itself. The use of radioisotopic labelled compounds allows to overcome this difficulty. The concentration of a labelled component in a multicomponent system is given by the radioactivity of a known amount divided by the specific activity of the labelled component.

If one of the components is labelled with ^{14}C and another one with ^{3}H, it is possible to measure, at the same time, the radioactivity of the two isotopes and consequently the concentration of two components. The method is not subjected to analytical limitations and, on account of the very high sensibility of the gauges, a quantity of 0.01 % can be measured in samples of about ten milligrams.

This procedure was tested by studying the separation of formic acid from acetic acid by means of continuous fractional distillation with different azeotropic mixtures. A column having a diameter of 20 mm and a length of 2200 mm, packed with Fenske rings, was employed. Four stainless steel needles were soldered in the glass wall, as shown in the figure; the needles were sealed by a teflon plug. The samples were collected by a microsyringe, which was progressively joined to the different needles.

Since the concentration of the azeotropic agent could be calculated from the temperatures along the column, what needed was the measurement of the change of the concentrations of formic acid, which was present in small amounts in the exhausting zone.

^{14}C labelled formic acid was employed (specific activity 4.10^{-3} $\mu C/mg$). The radioactivity was determined with a « Tricarb » liquid scintillation spectrometer; the samples, about 50 mg each, were neutralized with Hyamine in methanolic solution (scintillator composition : toluene, PPO, POPOP).

The lowest detected concentration of formic acid in acetic acid was 0.05 %. The time needed for reaching the equilibrium in the column, taking off and analyzing the samples did not exceed four hours.

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