

Automation of Wet Chemical Analysis with AMICA

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Zusammenfassung. Es werden neuartige Analysenautomaten für die industrielle Kontrollanalytik beschrieben: Ein Mikrocomputer steuert ein nach dem 'stopped flow'-Prinzip arbeitendes Dosier- und Mischgerät sowie ein Photometer und einen Probenwechsler. Diese Kombination erlaubt es, komplexere Algorithmen der Titrimetrie und Photometrie routinemäßig anzuwenden. Die Resultate unterschiedlicher Analysenmethoden werden mit etwa 0,2 % Standard-Abweichung in 1 bis 3 min erhalten.

Summary. New automatic analyzers for industrial control analysis are described: A microcomputer manages a liquid processing unit — working after the stopped flow principle — as well as a photometer and an autosampler. This combination allows to make use of complex algorithms for titrimetry and photometry in routine analyses. Analytical results are obtained from different methods with about 0.2 % S.D. in 1–3 min.

1. Introduction

During recent years a high degree of automation has been attained in standard clinical chemistry.

This is due to the following two facts: (I) Only a few — to a high extent closely related — methods had to be automated. (II) In many cases highly selective enzymatic reagents need only a few laboratory manipulations to be automated. The situation is quite different in industrial control analysis: there is a wide variety of different products, by-products, formulated chemicals and wastes, and because of this variety there are many different analytical procedures and usually only rather few samples at a time.

Attempts have been undertaken to automate these procedures. However neither the 'continuous flow' analyzers nor the 'batch' analyzers managed to cope with the variety of analytical procedures in industrial control analysis.

A novel approach to this problem is presented here: AMICA, an acronym for Automatic Modules for Industrial Control Analysis [1]. It automates the liquid sample preparation as well as measurement and result presentation. For solid sample preparation 'Pre-Set' [2] is used, which makes this system nearly universal.

In principle, the sample preparation unit is a stopped-flow device. This combines the advantages of the continuous flow

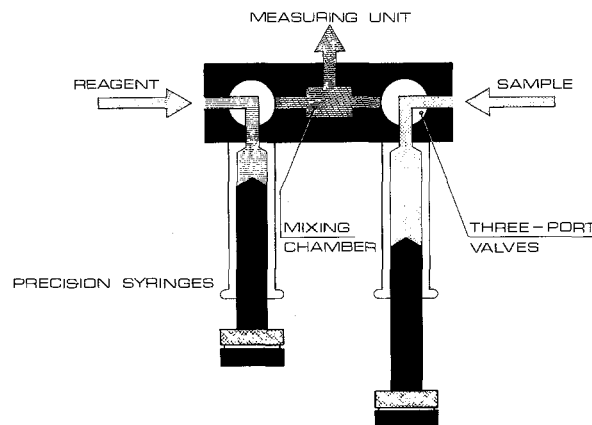


Fig. 1. Operating principle of AMICA liquid processing unit

methods: easy mixing, simple transportation on the one hand with the advantages of the discrete method: easy and repeated measurements on the other hand.

The complicated hydrodynamics of conventional segmented flow analyzers is replaced by the principle of positive displacement, whereas the complicated mechanics of batch-analyzers is omitted.

2. System Instrumentation

The liquid processing unit (LPU) consists of two step motor driven syringes with 3-way cocks (see Fig. 1). One syringe conveys the test solution, the other conveys the reagent, titrant or diluent. The outlets of the two syringes lead through a static mixing chamber to a measuring device: an electrode or a photometric cell. The degree of dilution is determined by the relative speed of the two step motors. The speed regulation is designed in a manner that the two movements always start at the same time and end at the same time: the piston with the longer distance therefore moves faster than the one with the shorter distance. As the speed does not change during a stroke, the degree of dilution at the outlet of the mixing chamber is constant.

Depending on the need of the analytical chemist, this liquid processing unit is combined with further peripheral instruments: An automatic photometric system originates, if a calculator and a photometer as well as an automatic sampler are added. (Fig. 2). Such a system is especially versatile if the calculator controls the wavelength via a RS 232 C data link. All kinds of photometric measurements may be carried out automatically: bichromatic measurement, multicomponent

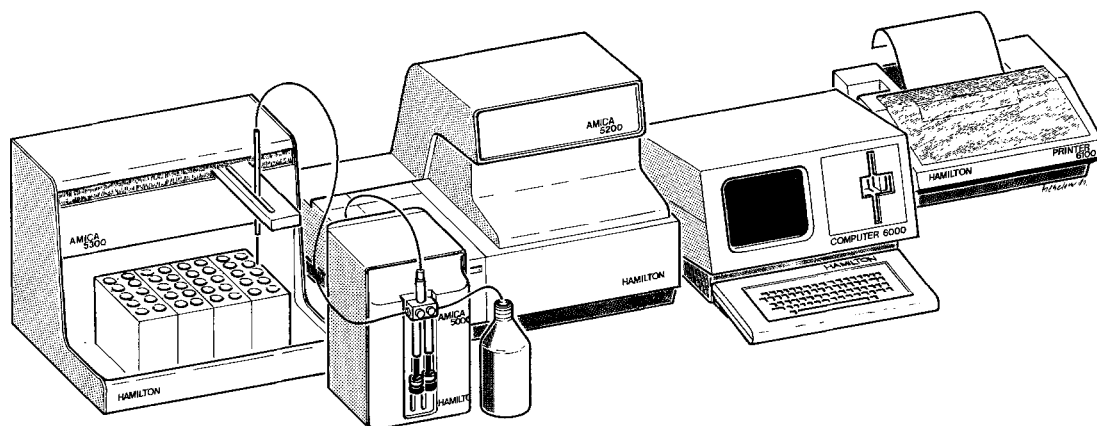


Fig. 2. AMICA system for photometric analysis and phototitration. It comprises an autosampler, the liquid processing unit, a spectrophotometer, a microcomputer and a printer/plotter

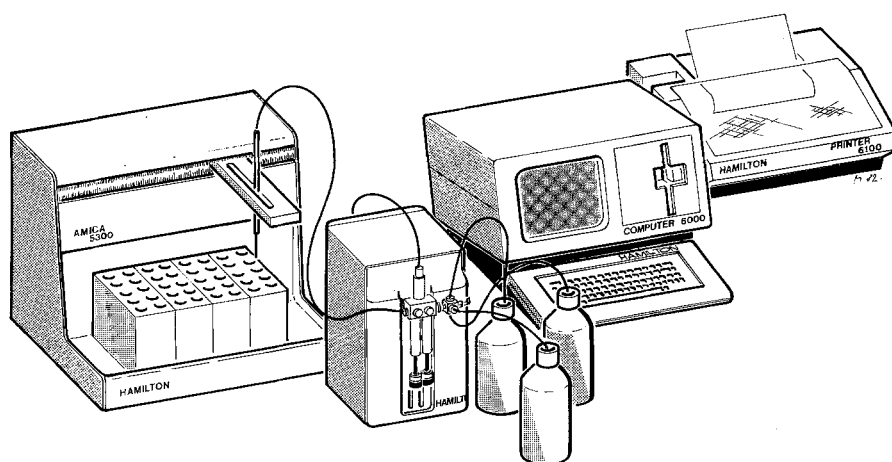


Fig. 3
The AMICA system for potentiometric titration comprises an autosampler, the liquid processing unit with an electrode, a microcomputer and a printer/plotter

analysis, kinetics, identity check of measured components. They will be discussed in detail under 'applications'. Of course, the photometry system is also suitable for direct and indirect photometric titrations.

On the other hand, there may be a combined electrode inserted directly into the mixing chamber. In this case, titrimetric analysis is possible in an unconventional manner: For each point of the titration curve a new mixture of sample and titrant is prepared and discarded after the measurement. The AMICA system for potentiometric titration is shown in Fig. 3.

3. System Software

Three software packages have been developed so far: photometry, potentiometric titration and phototitration. The programs are subdivided into four sections. The flowchart is displayed in Fig. 4.

The subroutine 'configuration' asks for the size of syringes at the right and left hand size of the instrument, for the speed of the burette movements (depending on viscosity and boiling point of the liquids), and other features. Tables 2, 5, 8 and 11 give examples.

The method section allows to create the analytical methods. It offers a choice of algorithms and evaluation criteria. Tables 1, 4, 7 and 10 give examples. The run section controls the analytical procedures until the result summary routine finishes the printout.

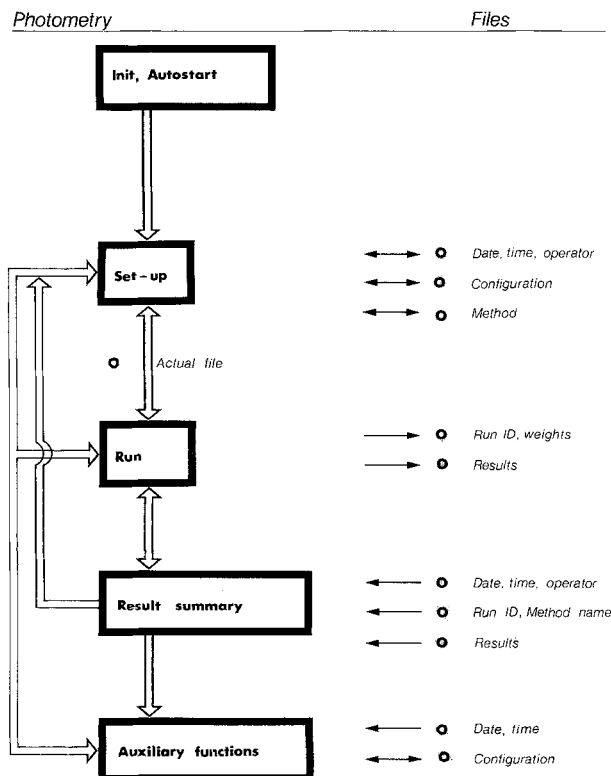


Fig. 4. Schematic illustration of AMICA system software organisation