I—24.

INTERFEROMETRIC AND CONDUCTOMETRIC DETERMINATION OF SOME BINARY MIXTURES BY KINETIC METHOD

V. VAJGAND and **T. TODOROVSKI**

Faculty of Sciences, University of Beograd, Institute of Chemistry, Technology and Metallurgy, Beograd, and Faculty of Technology and Metallurgy, Skopje

The reaction rate can be followed interferometrically, and by appropriate methods of evaluation of experimental data the composition of binary mixtures can be determined. The rate of reaction of acetic and propionic anhydride and mixtures thereof with water at 61.0 ± 0.1°C has been studied. In a modified interferometer cell the solution is vigorously stirred while being hermetically sealed at the same time.

For the sake of comparison, a conductometric method for the pure substances and their mixtures was developed, the measurements being made at the same temperature. In all the investigations, the initial concentration of anhydride in the solution was 10^{-3}M.

By the interferometric technique the composition of the mixture can be determined with an average error of ±3,5% (abs.), while by the conductometric technique it is ±3.6%.

Ketone mixtures can be interferometrically determined via the hydroxyl amine hydrochloride reaction only in mixtures of definite compositions, while the conductometric technique allows determination at any composition. Acetophenone and isobutyl methyl ketone were determined at 22.0 ± 0.1° in mixtures containing 30 to 70 percent of acetophenone, with an average error of ±1.4%.

I—25.

INTERFEROMETRIC VOLUMETRIC DETERMINATION OF CARBON DIOXIDE

T. TODOROVSKI, LJ. KECKAROVSKA, L. ŠOPTRAJANOVA and **I. SPIREVSKA**

Faculty of Technology and Metallurgy, University of Skopje

The interferometric volumetric determination of CO_2 is based on its absorption in a standard $Ba(OH)_2$ solution, taken in excess, and the titration of the excess with a standard solution of HCl.

The laboratory interferometer must be adapted for the titration as described in the paper "Interferometric Precipitation Titrations" *[Glasnik hem. društva Beograd, 34,* 261 (1969)*]*. Titrations were performed out of the interferometer cell in a vessel protected from atmospheric CO_2. The solution taken in the cell was thermostated at 22.0 ± 0.1°. For comparison a solution of NaCl was used with approximately the same refractive index as the solution

examined. The procedure for determination of CO_2 was developed on artificial mixtures containing 0.7 to 9.5 vol.% CO_2 with nitrogen. The mixture of gases was introduced into a 0.08 N solution of $Ba(OH)_2$ and the excess of the base titrated with a 0.3 N solution of HCl. The precipitate of $BaCO_3$ formed during the introduction of CO_2 did not interfere since it was filtered off during transfer of an aliquot of the solution to the interferometer cell. It is possible to determine CO_2 at below 0.7 %(vol.). In this case it is necessary to use three times more dilute solutions. The titration can be performed in the interferometer cell itself.

The results of titration are compared with those read directly from a calibration curve plotted from direct measurement of refractive indices on the interferometer. Differences are on the average not more than 0.1% abs. The method of determination from a calibration plot is much faster, but it requires precise preparation of mixtures of gases, which is time consuming. The titration method proposed is simpler, since easy to prepare solutions are used, and the titration is fast and accurate. Furthermore, this method is more selective, since by determinations of CO_2 using calibration plots other gases in traces can influence the accuracy of determination, while in titration method they practically do not interfere.

I—26.

ANALYTICAL MONITORING OF PYRIDOXINE (VITAMIN B_6) SYNTHESIS

J. LIPTÁK and **E. ULLRICH**

Pharmaceutical Chemistry Research Institute, Budapest

New methods have been developed and combined with the existing ones for analytical check in the manufacture of pyridoxine.

After the first step in synthesis, methoxyacetate is determined by gas chromatography in the presence of methyl chloroacetate. By ring closure 2-methyl-4-methoxymethyl-5-cyanopyridone-6 is obtained, identified by thin layer chromatography and measured by UV fluorescence. It is then nitrated and the product is determined by polarography in H_2SO_4. The polarographic behavior of the compound and the inhibiting effects of various substituents on the reduction was studied. By hydrogenation (in the synthesis), the aminomethyl product is obtained, contaminated with the cyanopyridine derivative. The ratio of these two compounds is determined by nitritometric titration with potentiometric end-point detection. After transformation of the amine to dihydroxyether, the product is condensed with 2,6-dichloroquinone chloroimide and determined spectrophotometrically at 650 nm.

The end-product (pyridoxine) is usually determined by titrating it in glacial acetic acid with $HClO_4$. But to eliminate interference from the preceding products, a selective method based on determination of the free hydroxyl group has been developed. Pyridoxine is complexed at pH 7 in the presence of borate buffer and the absorbence of the complex measured.

Since the dihydroxyether derivative has an anti-vitaminic effect, it is important to check the chemical and pharmaceutical purity of the end-product.