relatively stable, gave very erratic results with the various columns (see Table II), suggesting that it could not be efficiently resolved with the column packing that was used.

The aluminum block of the gas chromatograph was "preconditioned" with 100 μg quantities of the halogen compound to be tested, as suggested by the manufacturer of the instrument. The results of such treatment were not conclusive. Subsequently, the block was modified to include a quartz tube insert in the injection port area. However, since the vaporized sample component could still come in contact with sections of the metal block, this modification did not materially improve the degree of recovery of the compound.

Finally, the block and the columns were pretreated with tris-(2-biphenyl) phosphate[2] which has been suggested as an inhibitor of the Friedel-Crafts type of catalysis. Indiscriminate application of this reagent to the chromatographic instrument can produce adverse effects because of its low vapor pressure at 250°. Excess amounts may accumulate in the block and tangibly interfere with the analytical results. None of the above treatments materially improved the analytical results.

In conclusion, our experiences indicate careful sampling is necessary (accuracy is limited by the present type of syringes available), strict temperature control is essential, pin-hole leaks in the chromatographic systems must be avoided, and an all-glass or all-quartz system—including the sample injector area—seem to be preferable.

Pesticide Residue Research Laboratory, University of California, Davis, Calif. (U.S.A.) HERMAN BECKMAN ARTHUR BEVENUE

1 D. M. COULSON, *Technical Report No. II, Report No. 5*, Stanford Research Institute, Menlo Park, Calif., *SRI Project No. P-3198*, (1961) 7.
2 F. A. GUNTHER, R. C. BLINN AND G. K. KOHN, *Nature*, 193 (1962) 573.
3 D. D. PHILLIPS, G. E. POLLARD AND S. B. SOLOWAY, *J. Agr. Food Chem.*, 10 (1962) 217.

Received September 24th, 1962

J. Chromatog., 10 (1963) 231–233

An unusual ninhydrin reaction with inorganic cations

Ninhydrin, triketo-hydrindene hydrate, is universally used as a very sensitive reagent for amino-nitrogen in amines, amino acids and peptides.

ZACHARIUS AND TALLEY[1] reported recently that certain non-nitrogenous keto acids in eluates from ion exchange columns were found to react with ninhydrin to give coloured compounds. Although the colour thus obtained was mostly red, the danger of ascribing a colour reaction to the presence of certain nitrogenous compounds was pointed out. Nothing, however, is known about a reaction of ninhydrin with inorganic cations, except $(NH_4)^+$.

It was therefore greatly surprising to discover in a recent investigation of amino acids by high-voltage paper electrophoresis that some inorganic cations present in the sample produced a colour reaction similar to that of amino acids. Since under the chosen experimental conditions[2] the positions of K^+ and Na^+ almost coincided with those of $(NH_4)^+$ and dimethylamine, the identification of the latter compounds was thereby seriously complicated, if not entirely frustrated.

Fig. 1 illustrates the positions of several alkali and alkaline-earth metals with reference to $(NH_4)^+$ and those of some of the common amino acids, all revealed by application of a ninhydrin spray and heating for 10 min at 80°.

The reaction could only be produced on the paper, and not in solution. This might indicate a mechanism depending on certain impurities in the paper. Extracts from the

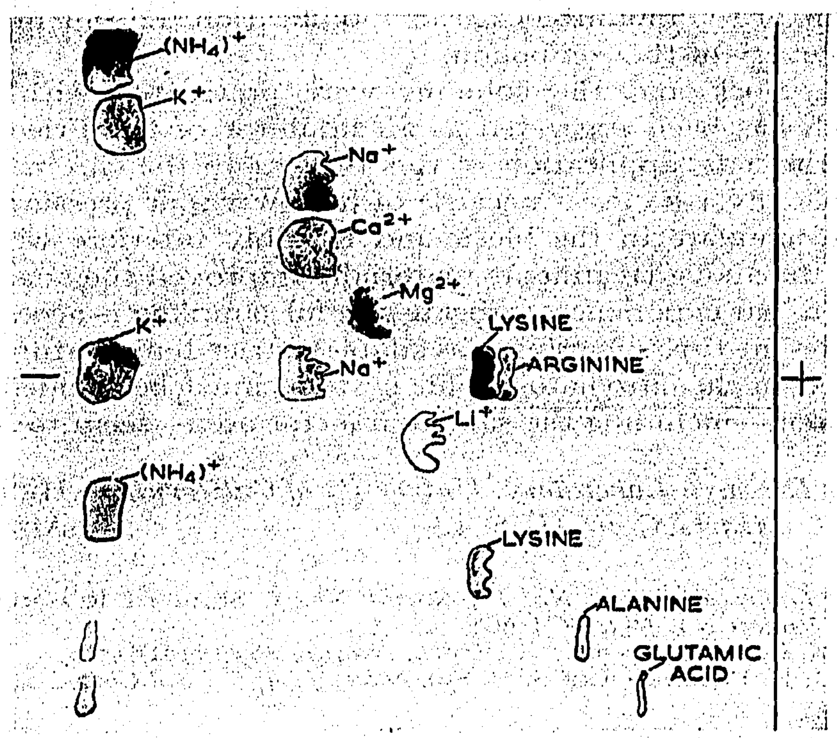

Fig. 1. An unusual ninhydrin reaction. Conditions: 100 V/cm, 6.7 mA/cm, pH 2.0, 0.75 *M* formic acid soln., Whatman No. 3 MM paper, 12°, 1.5 lb./sq. in., 15 min, spraying reagent = 0.1% ethanolic ninhydrin soln.

paper and the presence of shredded paper did not, however, produce a similar reaction in the test tube. The reaction worked on the paper strip not only at pH 2.0 but equally well with an electrolyte of pH 8.9 (ammonium carbonate solution).

Knowledge of this unusual reaction should lead to a more careful identification of $(NH_4)^+$ in the presence of K^+, and of dimethylamine in the presence of Na^+, but may also be utilized for the expedient detection of the presence of several inorganic cations such as K^+, Na^+, Li^+, Ca^{2+} and Mg^{2+} simultaneously with that of accompanying amino acids.

Tate & Lyle Research Laboratory,
Keston, Kent (Great Britain) D. GROSS

1 R. M. ZACHARIUS AND E. A. TALLEY, *J. Chromatog.*, 7 (1962) 51.
2 D. GROSS, *J. Chromatog.*, 10 (1963) 221.

Received October 24th, 1962