Impurities in analytical reagent grade chemicals

The rate of progress in chemistry is sensitive to the purity of the chemicals used in research work. In spite of the fact that many chemicals are sold as analytical reagent grade, it is discouraging to find that so many chemists need to purify chemicals further in order to ensure that the effects they are studying are not due to some impurity rather than the compound they are investigating. In our laboratory a working manual[1], on the purification of supposedly pure inorganic chemicals, has evolved during the years mainly through the painstaking work of DR. GEORGE BIEDERMANN and his associates.

The following experience with organic chemicals serves as an illustration of how hazardous it is to trust the labels on the bottles. In a program dealing with the principles of amine extraction, a technique useful in the reprocessing of nuclear fuels, we studied the extraction of water and nitric acid into xylene[2]. In Fig. 1 the results for water are given for two different bottles (I and II) of analytical reagent grade xylene from a well-known company. From Fig. 1 it is seen that the two samples

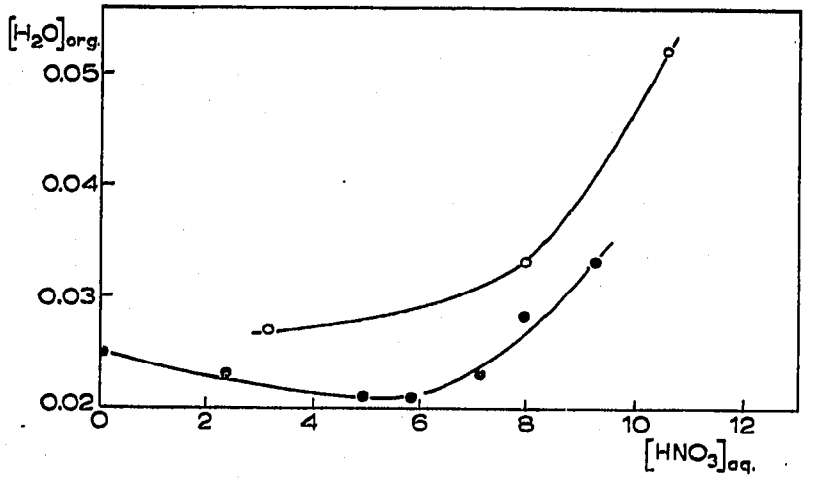

Fig. 1. The equilibrium molarity of water in the organic phase (determined by Karl Fischer titration) plotted against the equilibrium molarity of nitric acid in the aqueous phase for the system: xylene–HNO_3–H_2O. O = xylene I; ● = xylene II.

extract water quite differently. In order to check the purity of the two samples they were analysed by vapor phase chromatography (VPC). The results are shown in Table I, which shows that neither of the two samples meets with even very modest requirements of analytical purity. Samples as impure as these may be rare events;

TABLE I

VPC STUDY OF TWO DIFFERENT BATCHES OF XYLENE

Compound	*Constituent (%)*				
	m- + p-Xylene	*o-Xylene*	*Benzene*	*Toluene*	*Ethylbenzene*
Xylene I	89	6.7	0.6	0.4	3.2
Xylene II	59	21	—	1.1	18

unfortunately, however, it is not the first time the present writer has met with incredibly impure analytical reagent grade chemicals[3].

In order to prevent further experiences like these we now do a routine check on the purity of all volatile organic chemicals by VPC. However, not all research laboratories have access to a gas chromatograph. On the other hand the chemical industry can certainly afford to make routine checks by VPC in order to keep a uniform and high level of purity. Chemicals checked by VPC and labelled as gas chromatographically pure (to 99.5 % or better) would be of great help in scientific work. Although scientists constitute a small group of customers with strange and exclusive tastes, the feedback from research to industrial exploitation is of utmost economic importance. Guaranteed pure chemicals in the hands of the scientific workers would promote both speed and reliability in research work, to the advantage of all.

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[2] E. HÖGFELDT AND B. BOLANDER, *Report No. 4 to AB Atomenergi*, November 1961.
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A sample applicator for column chromatography

When using column chromatography it is important to have an even packing and an even level column surface. The column surface must not be disturbed when the solution of compounds to be separated is applied, otherwise horizontal bands will not be produced.

The sample is usually dissolved in the minimum amount of eluent in a small beaker and applied to the top of the column by allowing it to run from a pipette rotated rapidly around the inner wall of the column. For quantitative work, the beaker must be rinsed several times with the minimum amount of eluent and the rinsings applied to the column as described. This can be a tedious process and great care must be taken not to disturb the column surface.

The column head described (made in glass) avoids disturbance of the packing and the necessity of transfer of a solution in quantitative work.

The reservoir (A) (Fig. 1) is connected through the tap (B) to a tube (C). The latter passes centrally through a standard cone (D) which fits into a standard socket (E) at the top of the column. A small length of soft plastic tubing (F) is attached to the end of the tube which is bent as shown. The free end of the plastic tubing is just in contact with the wall of the column. After lubrication of the joint, the column