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## Note

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### Gas chromatographic determination of liquid loading in gas-liquid chromatographic packings

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In most physico-chemical measurements in gas-liquid chromatography (GLC) the volume of the liquid phase has to be accurately known. The amount of stationary phase is sometimes determined from weighings during the preparation of the packing<sup>1</sup> but usually it is determined from the weight of the support after combustion<sup>2</sup>, evaporation<sup>3</sup> or extraction<sup>4</sup> of the stationary phase. In this work a direct determination of the liquid phase loading is proposed by using a combination of Soxhlet extraction and GLC.

#### EXPERIMENTAL

Approximately 1 g of packing was weighed in a sintered-glass filter crucible and extracted with 80 ml of solvent for 4 h in a Soxhlet extractor. The amount of stationary phase in the solution was determined by GLC after dilution to 100 ml with the same solvent. Acetone was used as solvent for polar and *n*-hexane for non-polar and slightly polar stationary phases. Two stainless-steel columns, length 2.5 m and 1 m and I.D. 2 mm, packed with 5% SF-96 and 5% Dexil 400, respectively, on Chromosorb W AW DMCS (100–120 mesh) were used for the GLC determinations. Three injections were made for each sample and the peak heights from the sample and the corresponding standard were used for the quantification. The samples were injected with a microlitre syringe on to Varian 2700 and 3700 gas chromatographs equipped with flame-ionization detectors. Further experimental conditions are given in Table I.

#### *Test of accuracy and precision of the method*

The accuracy and precision of the method were tested with three samples, made by adding 25  $\mu$ l of dinonyl phthalate (DNP) with a micropipette directly on to 3.00 g of support in the sintered-glass filter crucible. The micropipette was rinsed with a small amount of the solvent (*n*-hexane), which also was adsorbed on the support. The same micropipette was used when preparing a standard solution of 25  $\mu$ l of dinonyl phthalate in 100 ml of *n*-hexane. Experimental results from this test are given in Table II.

The precision was further tested by comparing three 1-g samples from the same packing consisting of the support coated with a mixture of 10% dinonyl

TABLE I  
EXPERIMENTAL CONDITIONS

Stationary phase on Chromosorb W AW DMCS (100-120 mesh)	Liquid loading (% w/w)	GLC conditions	
		Column	Temperature
ODPN + DNP	9.97 + 9.47	5% SF-96	265°
ODPN	14.46	5% SF-96	180°
ODPN + octadecane	14.24 + 0.50	5% SF-96	180° for ODPN 230° for octadecane
OPDN + octadecane	13.62 + 4.44	5% SF-96	230° for octadecane
Squalane	1.01	5% Dexil 400	5 min at 80° 80-280° at 15°/min.
Squalane	5.00	5% Dexil 400	80-280° at 15°/min.
Di-(2-ethylhexyl) adipate	1.01	5% Dexil 400	5 min at 80° 80-300° at 12°/min'
Di-(2-ethylhexyl) adipate	4.95	5% Dexil 400	80-300° at 12°/min.

TABLE II  
TEST OF THE ACCURACY AND PRECISION OF THE METHOD

Sample	Liquid loading (%. w/w)
1	0.820
2	0.815
3	0.817
Standard	0.817

phthalate and 10% 3,3'-oxydipropionitrile (ODPN). Results from this test are given in Table III. The precision was also tested by extracting duplicate samples of packings with 5% squalane and 1% and 5% di-(2-ethylhexyl) adipate as stationary phases. The differences between the duplicate results were 0.52, 0.62 and 0.93%, respectively.

TABLE III  
TEST OF THE PRECISION OF THE METHOD

Sample	Liquid loading (%. w/w)		
	3,3'-Oxydipropionitrile	Dinonyl phthalate	Total
1	10.62	9.52	19.54
2	9.93	9.49	19.42
3	9.96	9.40	19.36
From weighings	—	—	19.46

*Comparison between the new method and a weighing procedure*

The determination of liquid loading from weighings during the preparation of the packing is one of the most reliable methods according to our experience, and it was therefore interesting to compare this method with the new procedure. For the different packings in Table I the difference in liquid loading determined with the two methods was generally less than 1%. This minor difference may be due to the limited

precision in the chromatographic determination. However, it seems reasonable, especially for low-loaded columns, that the difference in many instances may arise from two assumptions that must be made in the weighing procedure, namely that all of the weight losses during evaporation of solvent, drying and transfer of the packing to a weighing bottle are due to losses of stationary phase and that the weight of any adsorbed water can be neglected.

#### DISCUSSION

The results show that the accuracy and precision of the new method are very little influenced by the percentage liquid loading. This is in contrast to combustion and extraction procedures in which the need for a blank correction of the order of 0.3–0.6% of the weight of the support increases the uncertainty in the determination of the liquid loading of low-loaded columns. Further, no interferences arise due to losses of silanization and adsorbed water. The new method gives the liquid loading of each component in a mixture, which none of the other methods do. A knowledge of the loading of each component is especially important when preparing packings of relatively volatile stationary phases where the loss of stationary phase during evaporation of the solvent cannot be neglected. Consider, for example, the packing in Table III which was prepared by making a packing of 10.05% (w/w) of DNP dissolved in *n*-hexane, followed by evaporation of the solvent. The new method gives the additional information that the decrease of liquid loading from 10.05% (w/w) of each component to a total value of 19.46% (w/w) from the first weighings of support and stationary phase to the ready-made packing is mostly due to the loss of DNP. The method has hitherto been applied to volatile stationary phases with defined molecular formulae. With the use of liquid chromatographic methods it is also possible to determine silicones and other stationary phases of high molecular weight. However, the determination of non-volatile phases by liquid chromatography may in practice be less straightforward than that of volatile phases by GLC. In some instances it may be necessary to utilize the method of Wicarová *et al.*<sup>5</sup>, *i.e.*, weighing the amount of the stationary phase extracted from a sample of the packing.

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