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Analysis of internal filling gas of small-sized halogen lamps by gas chromatography

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ABSTRACT

The internal filling gas composition of small-sized (H-3) tungsten–halogen incandescent lamps was studied. An appropriate destructive sampling device was constructed and used to interface the sample source and the gas chromatograph. A high-resolution, sensitive chromatographic method was devised out for the determination of the main components in the lamp atmosphere and some of the impurities. It was found that decomposition products are formed inside the H-3 lamps during use. Their concentration as a function of the burn time can be followed by the proposed technique. The sampler interface with a Hewlett-Packard Model 5890A gas chromatograph and the isothermal elution method are described in detail. The concentration–burn time dependence gives information about the ageing process.

INTRODUCTION

The halogen lamp is a modern, highly efficient light source, the popularity of which is steadily growing. The main advantage of a halogen lamp, working at higher temperatures (2700–3100 K) than conventional Edison-type lamps, is that the inside of the glass bulb wall remains clear, even after a long burn time, and light-absorbing precipitated tungsten does not build up [1]. The halogen component of the internal filling gas is responsible for this beneficial property. The freshly prepared lamp atmosphere contains the main filling gas components (krypton and bromochloromethane) and some impurities.

During use, a variety of solid and gaseous products of the thermal decomposition reactions of the organic halide are formed and several of these can affect the lifetime of the lamp. In some instances the role of the different species in the life-shortening processes are still not completely understood.

EXPERIMENTAL

Sampling device and its interfacing with the chromatograph

The schematic design of the sampler developed for this work is shown in Fig. 1. The sampler is a 35-mm long stainless-steel cylinder of I.D. 12 mm, into which an H-3-type halogen lamp can be tightly inserted. Both ends are gas-tight, sealed with a metal switch-lock system. The air space can be evacuated down to $2 \cdot 10^{-3}$ mbar.

A screw-driven, stainless-steel breaker bar (1) serves for fracturing the glass bulb. A PTFE washer (2) provides a gas-tight seal. At both sides of the sampling device, inside the joining parts, there are fine filter disks (4) to protect the gas line from glass splinters. The insertion of the sample lamp and the cleaning of the chamber are accomplished through one end after detaching the sealing union nut (5).

Fig. 2 shows the layout of the measuring system used in these experiments. Through a pressure gauge and copper tubing, the gas chromatograph is supplied from a carrier gas cylinder (1). The above-described sampler (3) is attached to the sampling loop (6) of the chromatograph. Through two needle valves and a T-joint the sampling system can be evacuated by the rotary pump (5), the pressure being measured by the vacuum gauge (4).

Before introduction of the sample gas into the evacuated gas chamber, the sampling circuit is filled and emptied several times in order to avoid gas impurities.

Gas chromatography

The gas chromatographic analyses were performed on a Model 5890A gas chromatograph (Hewlett-Packard, Avondale, PA, U.S.A.) with a flame ionization detector for CH_4 , C_2H_6 , C_2H_4 , and CH_2ClBr , with a methanator and flame ionization detector combination for CO and CO_2 and with a thermal conductivity detector for H_2 . For the analysis of H_2 a $2 \text{ m} \times 1/8 \text{ in. I.D.}$ molecular sieve 5A (80–100

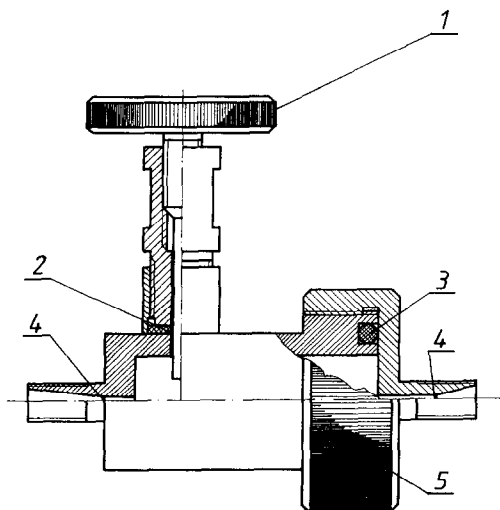


Fig. 1. Sampling device. 1 = Breaker bar; 2 = PTFE washer; 3 = Viton O-ring; 4 = filter disk; 5 = union nut.

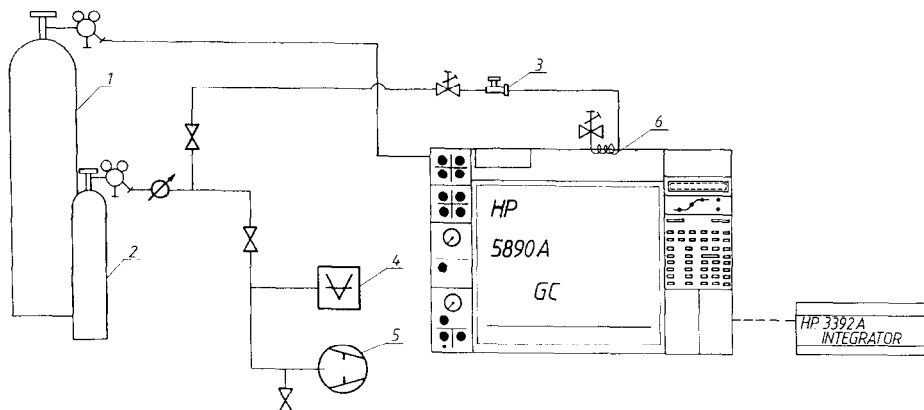


Fig. 2. Measuring system. 1=Carrier gas cylinder; 2=carrier or calibration gas; 3=sampling device; 4=vacuum gauge; 5=rotary pump; 6=sampling loop.

mesh), for CH_2ClBr a Porapak Q (80–100 mesh) and for CH_4 , CO , CO_2 , C_2H_4 , C_2H_6 a Porapak N (80–100 mesh) column were used [2,3]. Helium (6.0, Linde, Höllriegelskreuth, F.R.G.) and argon (5.0, Linde) were used as carrier gases at a flow-rate of 30 ml/min. The applied column temperatures were 40°C for the molecular sieve 5A, 60°C for the Porapak N and 120°C for the Porapak Q columns.

The calibration gases were obtained from Oxygen and Dissous-Gas (Budapest, Hungary) for quantification purposes.

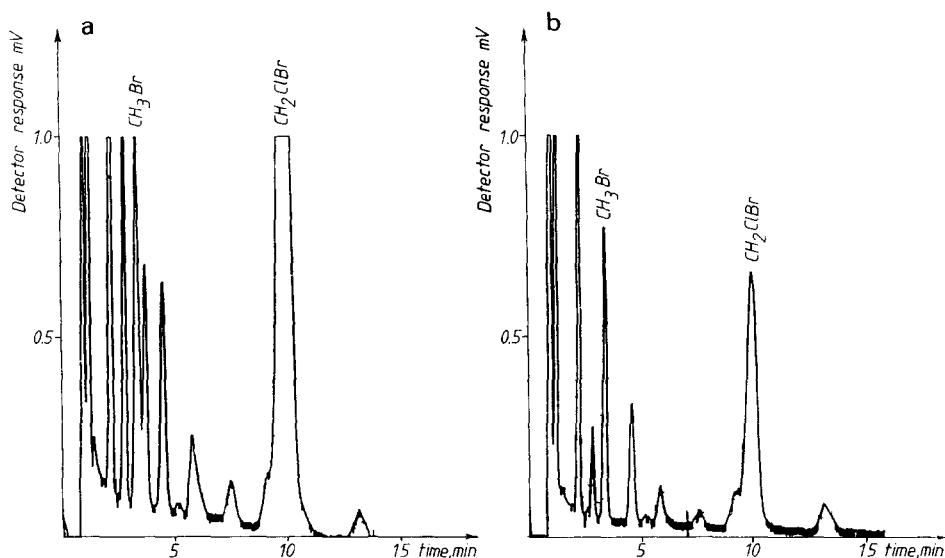


Fig. 3. Chromatograms of gas atmosphere of an H-3 lamp obtained with the Porapak Q column: (a) unused lamp; (b) burn time 1 s.

RESULTS

In order to obtain further chemical information about the gaseous products of the thermal decomposition reaction, specially prepared H-3 lamps, either unused or after certain burn times, were studied. The burn time was varied from 0 to 200 h and the concentrations of the main component (CH_2ClBr) and H_2 (as a characteristic decomposition product) were measured chromatographically.

Fig. 3a shows a chromatogram obtained from an unused lamp and Fig. 3b that from an H-3 lamp burned for 1 s. It is obvious that at a retention time of 10.4 min there is a significant difference between the two chromatograms, the peak which corresponds to CH_2ClBr being reduced after this short burn time. This indicates that CH_2ClBr is rapidly decomposed during operation of the lamp. After a burn time of 3 s, only traces of CH_2ClBr could be detected.

Interesting observations could be made when the dependence of hydrogen concentration on burn time was studied. Early in the lamp operation a rapid increase in hydrogen concentration takes place, which reaches a steady level in the gas phase after *ca.* 3 s and then slowly decreases over a long period.

The correct concentration of hydrogen helps to reduce the deposition of tungsten and thereby lowers the transport processes inside the lamps. Hence the correct hydrogen concentration is an important factor in increasing the lamp lifetime.

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