

Analysis of acetylacetone by means of gas chromatography

Since there is no method available in the literature¹⁻⁶ which is selective or sensitive enough for the analysis of acetylacetone, we have developed a gas chromatographic method for this purpose and have carried out gas chromatography on a crude, a purified and a pure acetylacetone sample. The investigations were carried out on a Carlo Erba Fraktovap Modell C type gas chromatograph. The following experimental conditions were found to be best:

Detector: thermistor; temperature 175.0°; bridge current 20 mA; sensitivity 3-50 %, depending on the amount of the sample.

Sample: 0.2-10.0 μ l.

Column: spiral of stainless steel; length 1800 mm; internal diameter 5 mm; packing 20 % silicone oil 550 on Celite (with particle diameter of 0.2 mm).

Temperature of the evaporator: 230°.

Carrier gas: hydrogen; flow rate 20 ml/min; pressure 0.13 kg/cm².

Temperature of the thermostat: 175.0°.

Compensograph: sensitivity 2.5 mV; paper speed 1.25 cm/min.

Fig. 1 shows the chromatogram of the crude acetylacetone. Using the internal standard method for qualitative identification and quantitative evaluation the presence of the constituents listed in Table I was established.

TABLE I
RESULTS OF GAS CHROMATOGRAPHIC ANALYSIS OF CRUDE ACETYLACETONE

No.	Constituent	Retention time (min)	Retention volume (ml)
1	Acetoacetic ester (contamination)	1.28	25.60
2	Acetone	1.84	36.80
3	Diacetone alcohol	2.00	40.00
4	Acetic acid	2.16	43.20
5	Ethyl acetate	2.32	46.40
6	Water	3.02	60.40
7	Acetylacetone	3.92	78.40
8	Phorone	6.48	129.60
9	Acetyl-acetoacetic ester	10.55	211.00

In the acetylacetone purified by distillation only the following constituents were found: (1) Acetoacetic ester (contamination), (2) diacetone alcohol, (3) acetic acid, (4) acetylacetone, and (5) phorone, while the pure acetylacetone contained only the following trace impurities: (1) acetoacetic ester (contamination), (2) diacetone alcohol, and (3) acetic acid.

Comparing the data obtained from the analysis of the crude, purified and pure acetylacetone respectively, it is clear that similar mixtures were analysed, only the number and the amount of the contaminants differing in the samples.

An interesting phenomenon has been observed with chromatography of pure acetoacetic ester, when seven peaks were observed (see Fig. 2). After many measurements we established that the first peak is caused by contaminations, while the others are due to the acetoacetic ester. They are probably the consequence of equilibrium

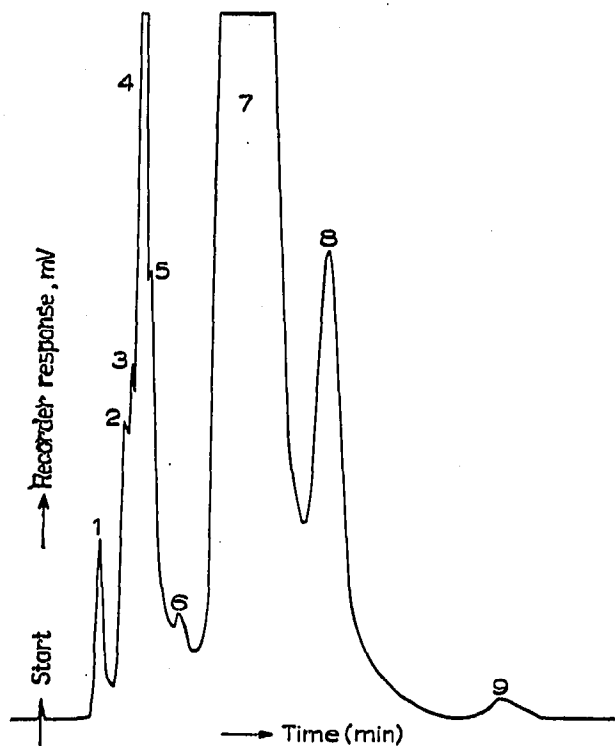


Fig. 1. Chromatogram of purified acetylacetone.

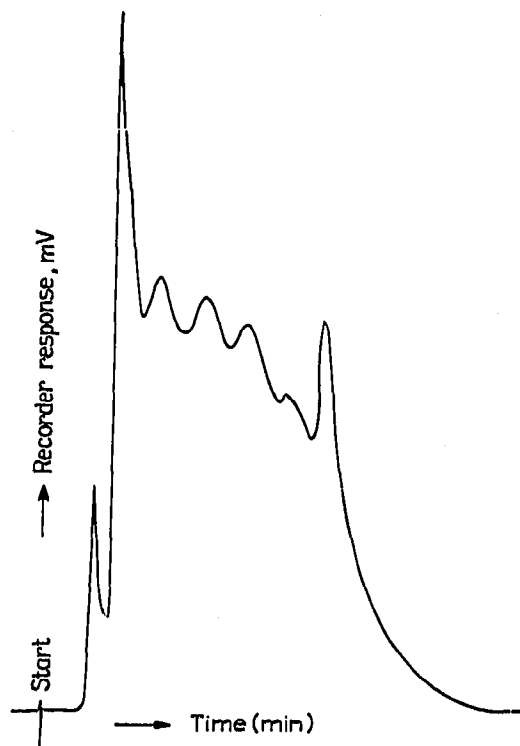


Fig. 2. Chromatogram of pure acetoacetic ester.

positions of keto-enol rearrangement and internal transformations of acetoacetic ester during analysis. Experiments to elucidate this phenomenon are in progress and results of these studies will be published later.

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