Rapid Procedure for the Determination of Residual Acetone and 2-Propanol in Oilseed Meals and Flours[1]

H.P. DUPUY, E.T. RAYNER, and **S.P. FORE,** Southern Regional Research Center, ARS-USDA, New Orleans, Louisiana 70179

ABSTRACT

A simple, rapid elution gas chromatographic method was used for quantitative determination of acetone or 2-propanol in finely ground, extracted oilseed meals and flours. In the procedure, a 0.05 g sample of oilseed meal or flour was placed between 2 small glass wool plugs in a liner of the injection port of a gas chromatograph. Water (250 μl) was added over the glass wool sandwich, and the liner was placed in the heated injection port and firmly secured. Acetone or 2-propanol and other volatiles were rapidly eluted from the sample onto the column by the combined action of heat, moisture, and carrier gas. The components then were resolved by temperature programmed gas chromatography (GC) on a 1 ft x 1/4 in. Porapak Q column. A second gas chromatographic method, a volatilization procedure, was included in the study for comparison as a measure of the concentrations of acetone or 2-propanol. Samples of oilseed meals and flours were analyzed by both methods. The rapid elution procedure proved to be a simple and effective means of detecting residual acetone or 2-propanol in the samples. The values were very similar to those obtained by the volatilization procedure.

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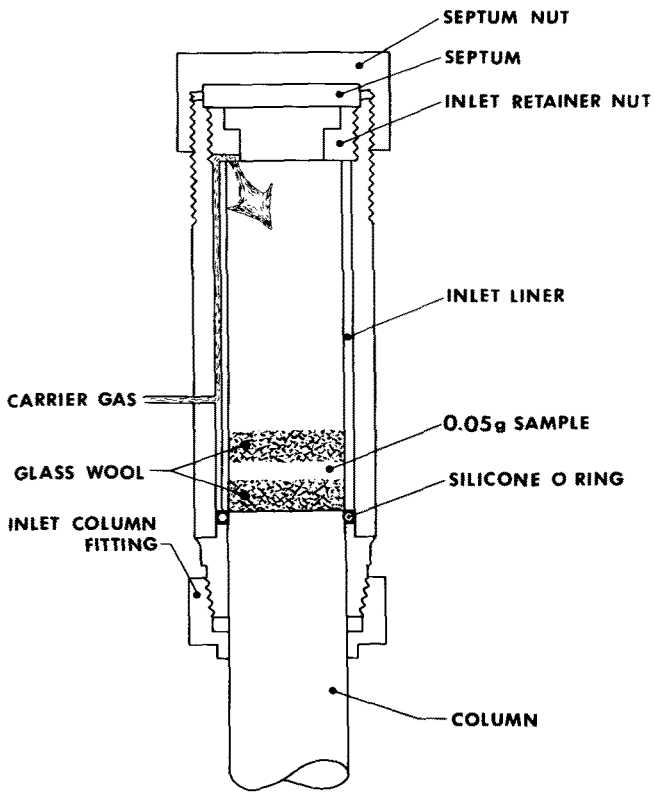

FIG. 1. Cross section of inlet of gas chromatograph showing inlet liner with sample.

INTRODUCTION

Acetone and 2-propanol have been reported to be effective solvents for the extraction of aflatoxins from contaminated cottonseed or peanut meals (1,2). When such commodities are processed with these solvents, however, it is essential to quantitatively determine the levels of residual solvent in the finished product. A number of procedures have been developed for the determination of residual solvents in extracted agricultural products such as oilseed meals, but none of them is rapid enough to monitor modern continuous desolventization processes. The flash cup method for hexane (3) for example, is time consuming and not effective below a concentration of 300 parts per million (ppm). the azeotropic distillation procedure (4), applicable to the detection of various solvents, is very sensitive, but involves much work and the use of a correction factor which has a tendency to vary with different types of samples. The volatilization procedures (5-7) are very simple and sensitive, but are also time consuming, requiring as much as 5 hr to volatilize residual acetone from samples of oilseed meals or flours prior to gas chromatographic analysis of the headspace gas (6). Although the volatilization procedures may be useful for routine determinations where time is not a factor, they are less suitable for applications where rapid analysis is necessary. This paper describes a simple, rapid, sensitive, gas chromatographic method for the quantitative determination of residual acetone or 2-propanol in extracted oilseed meals or flours, and compares the method with a gas chromatographic volatilization procedure applicable for the detection of these solvents.

EXPERIMENTAL PROCEDURES

Materials

The following products were used: Porapak Q (a porous polymer), 80-100 mesh, (Waters Associates, Inc., Framingham, MA); Silicone O rings (preconditioned at 200 C for 2 hr to render them free of volatile impurities) (Applied Science Laboratories, Inc., College Station, PA); Pyrex brand glass wool (Corning Glass Works, Corning, N.Y.); and acetone and 2-propanol, analytical reagent grade, (Mallinckrodt Chemical Works, St. Louis, MO.).

Sample Preparation

A small plug of glass wool was placed in the end of the liner (3 5/16 in. length of 3/8 in. outer diameter [OD] borosilicate glass tubing) of the injection port of the gas chromatograph. The glass wool was tapped down lightly and 0.05 g finely ground oilseed meal or flour was added on top of it. The sample was capped with another small plug of glass wool. Water, (250 μl) was added on the top layer of the glass wool-oilseed meal sandwich and allowed to moisten the sample for ca. 5 min. The liner, containing the moistened sample and glass wool, was inserted on top of the silicone O ring in the preheated (95 C) injection port of the gas chromatograph. The inlet retainer nut then was tightened firmly to form a seal between the inlet liner and the base of the injection port. The septum was placed in position and tightened securely with the septum nut; thus the carrier gas was forced to flow upward and into the liner. Fig. 1 shows construction of the complete inlet liner assembly. Thus prepared, the sample was analyzed by direct

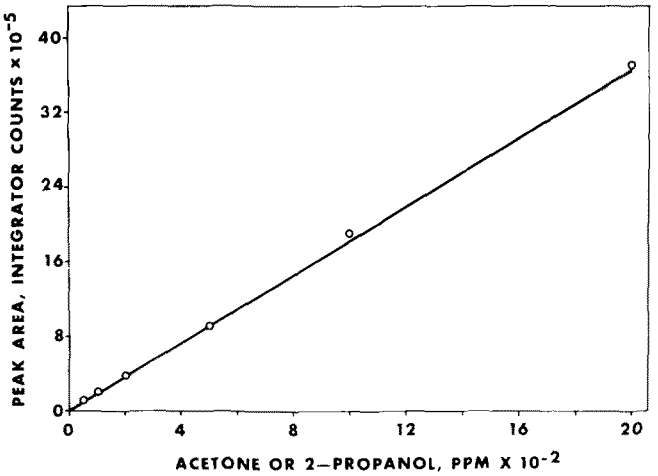

FIG. 2. Direct gas chromatograph calibration plots for converting peak area integrator counts to ppm of acetone or 2-propanol.

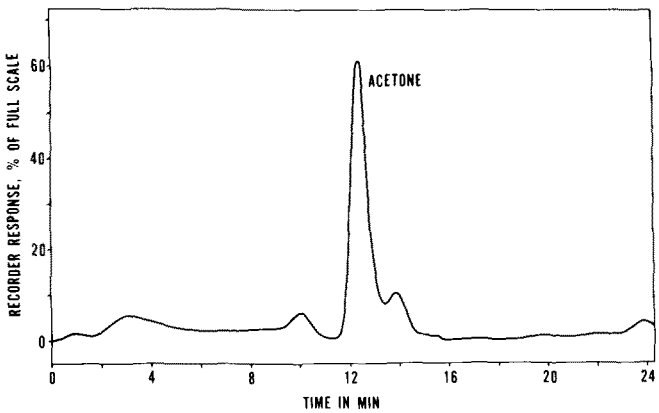

FIG. 3. Representative curve from the rapid elution procedure for a sample of cottonseed meal containing 20 ppm acetone.

GC as described later. When the temperature programming was complete and the acetone or 2-propanol peak had been completely eluted, the spent sample was removed from the inlet, and 20 µl water was injected to clean the column during the final hold period.

Gas Chromatography

The following GC conditions were employed for rapid elution of residual acetone or 2-propanol from finely ground oilseed meals and flours and subsequent resolution of the respective peaks. Instrument: MicroTek 2000 MF with dual independent hydrogen flame detectors. Recorder: Westronics LD 11 B. Integrator: Infotronics CRS-100. Columns: 1/4 in. OD stainless steel U-tubes, 1 ft long packed with Porapak Q (80-100 mesh). Carrier gas: helium. Flow rates: helium, 60 ml/min each column; hydrogen, 52 ml/min to each flame; air 1.2 cu ft/hr (fuel and scavenger gas for both flames). Temperature: detector at 200 C; injector port at 95 C; columns programmed between 60 and 190 C; initial hold at 60 C for 5 min; programmed at 5 C/min for 26 min; final hold at 190 C for 20 min. Attenuation: 10 x 1 and Auto x 1 for integrator. Chart speed: 30 in/hr.

Standardization

A calibration curve for use with a 0.05 g sample of finely ground oilseed meal or flour was prepared as follows. A micro syringe was used to add 0.02 g acetone to 20 g water to yield an aqueous solution containing 1000 ppm acetone. The same procedure with 2-propanol was used to prepare a solution containing 1000 ppm 2-propanol. A portion (10 g) of each solution was removed and labeled for subsequent use. Then, 1 g of each of the remaining original 1000 ppm solutions was diluted separately with 9 g water to provide 2 additional solutions, one containing 100 ppm acetone and another containing 100 ppm 2-propanol. Aliquots (25 µl, 50 µl, and 100 µl) of the 4 standard solutions containing 100 ppm and 1000 ppm of each solvent, then were analyzed by direct GC, and the values were plotted against peak area counts. The procedure for the gas chromatographic analysis of these standard solutions was the same as that described previously, with the exception that the glass liner contained only glass wool, and the aliquots of solution were injected through the septum with a GC syringe to preclude any possibility of solvent loss by evaporation. The calibration chart constructed in this manner is shown in Figure 2.

TABLE I

Determination of Residual Acetone by Volatilization and Rapid Elution Procedures

Meal sample	Acetone recovered (ppm)	
	Volatilization	Rapid elution
Cottonseed	6	6
Cottonseed	15	10
Cottonseed	20	20
Cottonseed	130	120
Cottonseed	550	510
Cottonseed	1600	1550
Cottonseed a)	25	25
Cottonseed a)	45	45
Cottonseed a)	200	200
Soybean	5	5
Soybean	6	8

a) flour samples.

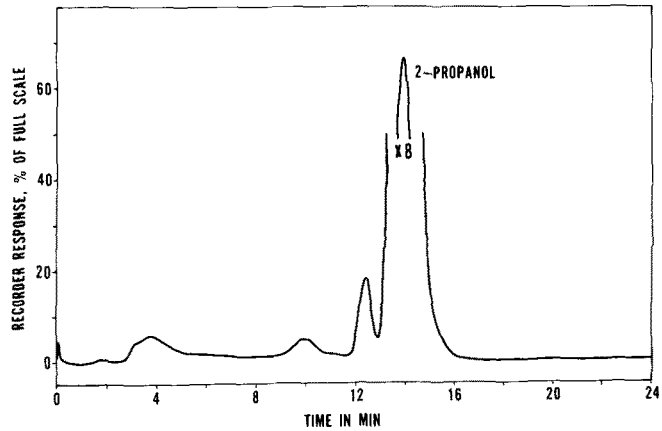

FIG. 4. Representative curve from the rapid elution procedure for a sample of cottonseed meal containing 230 ppm 2-propanol.

Volatilization Procedures

The volatilization procedures used to obtain the comparison values for acetone and 2-propanol content of oilseed meals and flours shown in Tables I and II have been described previously (6,7).

RESULTS AND DISCUSSION

The elution of residual acetone and 2-propanol from oilseed meals and flours by direct GC is accomplished by the interaction of 3 variables, heat, moisture, and carrier gas. An operating temperature of 95 C appears to be optimum for volatilizing the components within a reasonable time with minimal decomposition of the sample. In acetone analysis, it is essential to maintain the lowest practical operating temperatures, as it has been shown (6) that traces

TABLE II

Determination of Residual 2-Propanol by Volatilization and Rapid Elution Procedures

Meal sample	2-Propanol recovered (ppm)	
	Volatilization	Rapid elution
Cottonseed	10	10
Cottonseed	15	15
Cottonseed	25	25
Cottonseed	210	230
Cottonseed	750	780
Cottonseed	1650	1600
Soybean	ND[a]	ND
Soybean	ND	ND
Fish	15	20
Fish	390	420

[a]ND = None detectable.

of acetone may be generated in oilseed meals which are heated to high temperatures.

The presence of adequate moisture is a critical factor in the release of residual acetone or 2-propanol, which, in some instances, may be strongly bound in the sample material. Heating alone, with insufficient moisture will not release the residual solvents. A proportion of 5 parts water to 1 part oilseed meal, by wt, was effective for releasing the solvents, which were then readily swept onto the chromatographic column by the flow of carrier gas.

The sensitivity of the rapid elution method of analysis for acetone is illustrated in Figure 3. The representative GC peak shown for acetone was derived from the analysis of a cottonseed meal containing 20 ppm residual acetone.

A comparison of the results from the analysis of residual acetone in cottonseed and soybean meals by the volatilization (6) and the rapid elution procedures is shown in Table I. Values by the 2 methods are identical for essentially half of the samples, and agreement among the remaining values is quite good. No values below 5 ppm are indicated for either method. This may be due, in part, to the tendency of oilseed meals to generate traces of acetone on heating.

Figure 4 shows a representative GC curve obtained by rapid elution for a sample of cottonseed meal containing 230 ppm residual 2-propanol.

Table II compares the results of volatilization (7) and rapid elution analysis for 2-propanol in various samples of oilseed meals and fish flours. In half of the examples shown, the values are identical. The remaining results agree quite well with only a slight tendency toward higher values for those samples analyzed by rapid elution. The excellent agreement of the methods at the more meaningful lower levels suggests a specific advantage for the rapid elution procedure because it is inherently simpler and considerably faster. The rapid elution technique should be especially useful for monitoring production operations where many samples must be analyzed in a minimum of time.

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