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Hexane Losses in Solvent Extracted Soya Meal: Measurement by Gas Chromatography and Brief Evaluation

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ABSTRACT

Hexane losses in solvent extraction plants arise from a variety of causes. With the constantly increasing price of hexane, it has become imperative to identify the major loss areas and quantify losses in these areas. Hexane lost in meal leaving the desolventizer-toaster (D-T) is an obvious choice for investigation. A method has been developed for determining relatively high levels of hexane in hot meal leaving the D-T. The method was designed to minimize the loss of solvent by evaporation between the time of sampling and the actual measurement by gas chromatography (GC). Examination of the results confirmed that losses of hexane as a result of inadequate stripping of the meal in the D-T formed a very significant part (10–40%) of total hexane losses.

INTRODUCTION

A modified volatilization procedure has been described (1) which overcomes some of the possible deficiencies in earlier gas chromatography (GC) methods developed by Dupuy and Fore for determining residual hexane in meal (2, 3). In essence, all these methods have been developed to provide a further degree of quality control over the final product meal.

There still exists, however, a need for a method which will determine hexane in meal in the 500–4,000 ppm level actually encountered in practice when solvent extraction plants are being run at or above rated capacity. The loss of hexane from the hot, moist meal leaving the D-T is minimized in the method described here by the following procedures: (a) initial sampling is done rapidly into a container which is sealed when full; (b) solvent extraction with cyclohexane, using relatively large aliquots (50 g meal to 100 ml solvent) is used to extract the hexane. (For complete extraction, a 24-hr steeping period was found to be necessary). A further feature of the method, apart from its relative simplicity, is that no modifications are required to GC equipment.

EXPERIMENTAL PROCEDURE

Sampling

A 1-kg plastic container equipped with screw-top lid was inserted into a connecting chute of the meal conveyor system leaving the D-T and was closed as soon as it was full. Meal sampled in this way contained 13–14% moisture and all hexane measurements were subsequently related to

TABLE I

Recovery Data

Recovery (%)	Initial spiking (ml)	Known additions (ml)	Peak Height (adjusted for cyclohexane impurities) Line counts
95	50	0	4.5
		250	30
		500	58
		750	86
97	100	0	8
		50	14.2
		100	19.2
		150	23.0
		175	25.0
		200	27.5
97	150	0	13.5
		50	17.0
		100	21.1
		150	25.2
		200	30.4
95	200	0	16.0
		50	20.2
		100	24.7
		150	30.0
		200	35.8

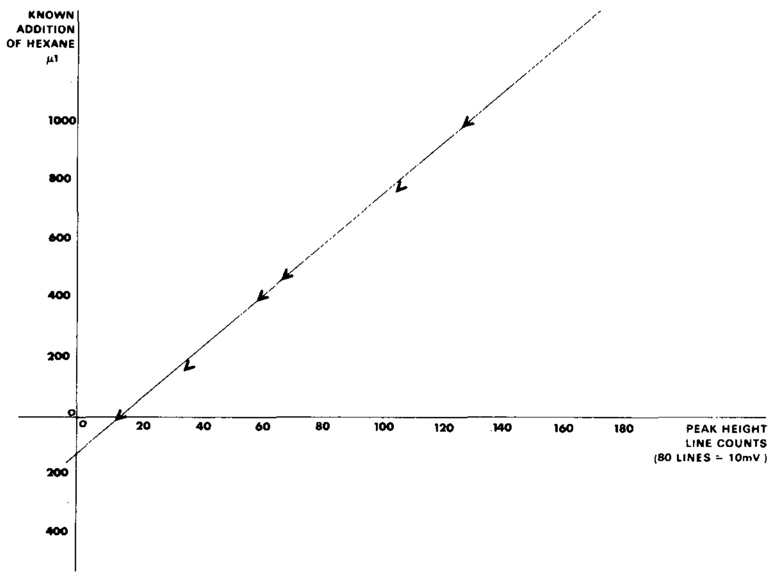

FIG. 1. Determination of residual hexane in meal by standard additions—typical result for meal containing 0.24% v/w hexane.

meal at this moisture content.

Extraction

A 50-g aliquot of the sampled meal was weighed rapidly into a 400-ml Erlenmeyer flask and 100 ml Cyclohexane AR was added immediately. A rubber bung, protected with aluminium foil, was inserted and the flask was allowed to stand for 24 hr.

Gas Chromatography

A dual column Pye-Unicam gas chromatograph. Model GCD, equipped with twin FID detectors, was used. (Since temperature programming was not required, only one

TABLE II

Meal Hexane—Typical Results

Sample	%, v/w	Meal hexane as % of total hexane loss
Day 1	0.24	30
↓ 2	0.32	40
3	0.14	17.5
4	0.16	20
5	0.12	15
6	0.26	32.5
7	0.20	25

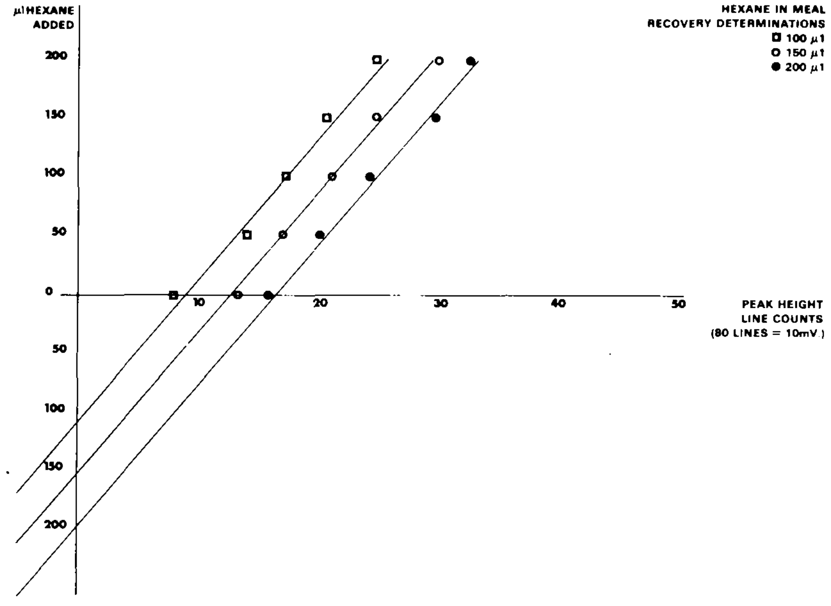

FIG. 2. Recovery determinations on spiked samples containing 100, 150, and 200 µl hexane per 50 g meal.

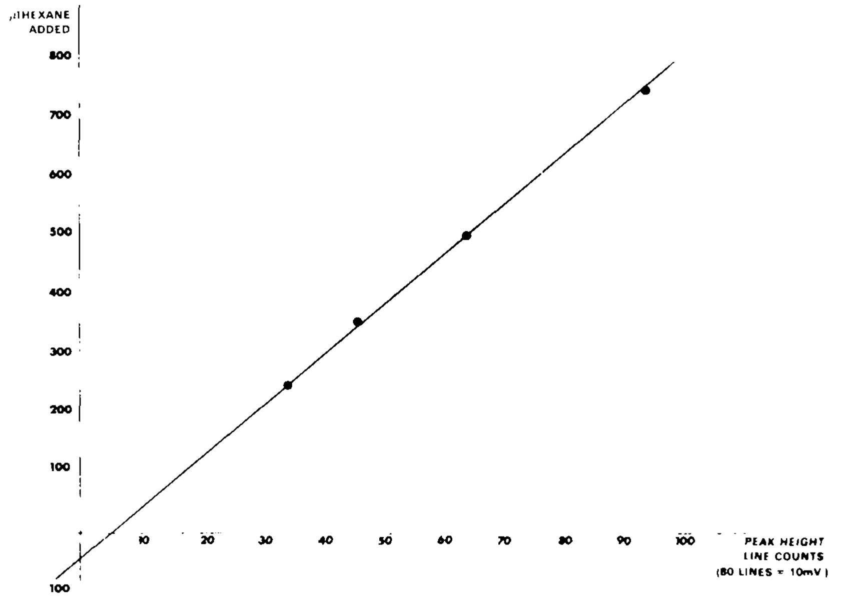

FIG. 3. Recovery determination spiked sample containing 50 μl hexane per 50 g meal.

column was used). The recorder was a Perkin-Elmer-Hitachi flat-bed recorder with disc integrator. Columns were glass and 1.5 m long and 4 mm id, packed with 10% squalane on Diatomite CQ 100-120 mesh. Operating conditions were: carrier gas flow—nitrogen, 50 ml/min; hydrogen flow, 55 ml/min; air flow, 800 ml/min; injector temperature, 100 C; column temperature, 75 C; detector temperature, 150 C; attenuation, 16 x 10 and 32 x 10; sample size 1 μl.

Cyclohexane AR grade was first injected to ascertain impurity levels. This was followed successively by 1.0 μl samples from the cyclohexane extract before and after spiking with μl aliquots of hexane to a total of 1,000 μl. A graph was then constructed of peak height for the hexane peak (x axis) vs known addition of hexane (y axis). Doubts about the reproducibility of μl injections were overcome by using 4 known additions to give a total of 5 points for each graph—found to be essentially linear in every case.

The (negative) intercept of the straight line graph on the y axis gave the value of the hexane, in μl, contained in 50 g meal (Method of Standard Addition, Fig. 1). Multiplication by a factor of 2 x 10^{-3} gave percent hexane expressed by vol on mass of meal (ml hexane/100 g meal).

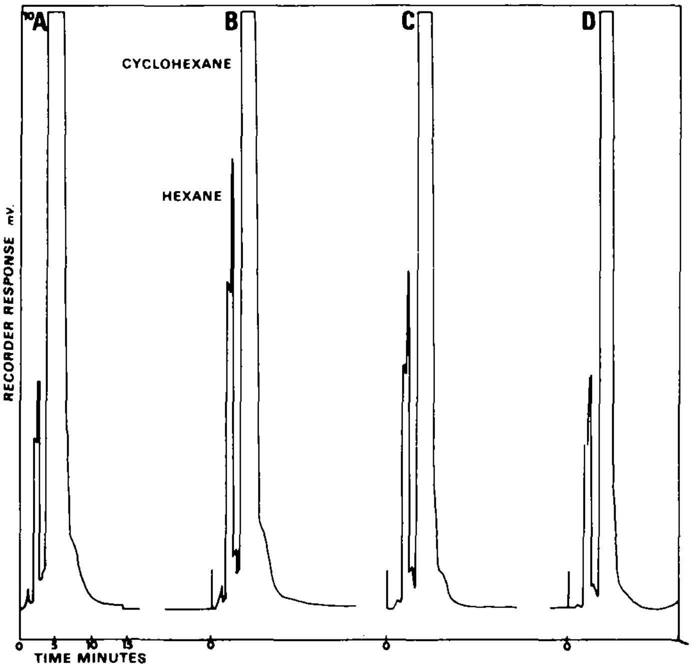

FIG. 4. Typical hexane determination by standard additions. Gas chromatograms of cyclohexane meal extract: (A) 250 μl addition, attenuation 16 X 10; (B) 500 μl addition, attenuation 16 X 10; (C) 750 μl addition, attenuation 32 X 10; (D) 1,000 μl addition, attenuation 64 X 10.

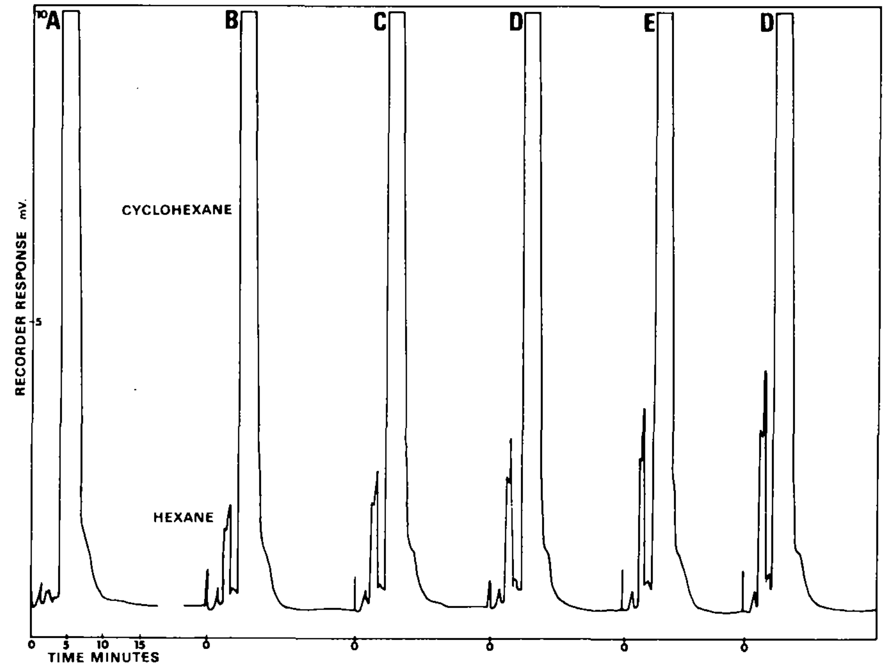

FIG. 5. Recovery determinations. Gas chromatograms of: (A) oven-dried, unspiked meal extracted with cyclohexane; (B) sample (A) spiked with 150 μl hexane–cyclohexane extract; (C), (D), (E), (F) sample (B) with successive additions, in total, of 50 μl, 100 μl, 150 μl, 200 μl hexane–cyclohexane extracts. Attenuation 16 × 10 throughout.

RECOVERY DETERMINATIONS

A hexane free meal sample was prepared by oven drying meal at 105 C for 5 hr. Portions of meal (43-g) weighed out into stoppered 400 ml Erlenmeyer flasks were then treated with 7 g distilled water to produce 50 g portions of meal at 14% moisture content.

These samples were then spiked with varying μl quantities of hexane in the range 50–200 μl and subjected to hexane determination as described (Table I, Fig. 2 and 3). This range was selected because it corresponded to the range of hexane in meal values encountered in practice, i.e., 0.10–0.40% v/w. Inevitably, under-recovery was observed but 95–96% recovery was considered entirely acceptable for the purpose of monitoring major hexane loss areas. The lower limit of detection by this method may be regarded as the level of hexane impurity in cyclohexane AR, e.g., 0.01% (100 ppm) maximum.

RESULTS AND DISCUSSION

Some typical results are given in Table II. In the last column, the meal losses have been expressed as a percentage of total plant losses, for which a monthly average figure of 0.8% v/w was used, again expressed on a meal basis.

Clearly, hexane losses in meal form a very significant part of the total process losses. This has led to an investigation on ways to improve D-T operation. Alternative designs of D-T which have been reviewed recently also are under consideration (4).

In conclusion, it should be said that the emphasis here on hexane losses does not detract from the overall objective of reducing unit costs by efficient plant operation. An excellent summary of the main factors affecting throughput and performance was published by Myers (5) in which hexane losses were actually not highlighted as being a major problem. This may still be true today for plants in the U.S., but for countries such as Zimbabwe, without indigenous petrochemical production, the cost and the consumption of hexane are major factors to be considered.

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