GASOLINE

Contents

DETERMINATIVE METHOD

A. INTRODUCTION

Theory

The canned product is cooled in a freezer before opening, because canned meats do not usually have enough headspace for conveniently taking a saturated vapor sample. Fat is transferred to a 10 mL glass vial, which is then sealed with aluminum foil and capped with a Teflon-coated rubber septum and aluminum cover. The vial is heated at 100° C for 15 minutes and a 100 μL injection is made using a gas-tight syringe on a gas chromatograph equipped with a flame ionization detector (FID).

DETERMINATIVE METHOD

B. EQUIPMENT

1. Apparatus

a. Nitrogen, hydrogen, and compressed air sources: At 20 mL/min (or 80 mL/min, if glass column is used), 10 mL/min, and 100 mL/min respectively.

b. Glass vials: 10 mL, with suitable septums and covers.

c. Gas-tight syringe: 100 μL, Hamilton (1710-N), or equivalent.

d. Syringes: 10 μL and 100 μL.

e. Forced draft oven, or other heating device: Set at 100° C.

f. Volumetric flask: 10 mL.

2. Instrumentation

a. Gas chromatograph: Hewlett-Packard Model 5830, or equivalent, equipped with a flame ionization detector.

b. Stainless steel column: 6′ × 4 mm i.d. glass column (80 mL/min N_2 flow rate), packed with G.P. 5% SP-1200/1.75% Benton 34, on 100/120 mesh Supelcoport.

DETERMINATIVE METHOD

C. REAGENTS

Reagent List

a. Gasoline: regular or unleaded.

b. Petroleum ether: pesticide grade.

c. Ortho xylene: reagent grade.

d. Meta xylene: reagent grade.

e. Para xylene: reagent grade.

f. Ethyl benzene: reagent grade.

DETERMINATIVE METHOD

D. STANDARDS

1. Source	Gasoline: purchase from local source, regular or unleaded. Each new lot of standard should be prepared as stated in the methodology and compared to current standards.
2. Preparation of Standards	Working standards. i. Gasoline—1:10, gasoline/petroleum ether, V/V. ii. Ortho xylene—1:10, o xylene/petroleum ether, V/V. iii. Meta xylene—1:10, m xylene/petroleum ether, V/V. iv. Para xylene—1:10, p xylene/petroleum ether, V/V. v. Ethyl benzene—1:10, ethyl benzene/petroleum ether, V/V.

DETERMINATIVE METHOD

E. EXTRACTION PROCEDURE

1. Sample Preparation

Place canned meat sample in a freezer for one hour.

2. Sample Extraction

a. Transfer approximately 1 g fat from the surface of the solidified meat mass inside the can to a 10 mL glass vial.

b. Seal the vial with aluminum foil, a Teflon-coated rubber septum, and the aluminum cover cap, in that order.

c. Run 1 ppm and 10 ppm standards (approximations only) in parallel with sample by adding 10 μL and 100 μL respectively of 1:10 gasoline/petroleum ether to 1.0 g clean fat in each of two vials and performing steps d and e below.

d. Heat the sample in the sealed vial in a forced draft oven at 100° C for 15 minutes. If heating block with holes that fit vials is substituted for oven, sample can be taken for step e below while vials remain at 100° C.

e. Using a 100 μL gas-tight syringe, inject a 100 μL headspace sample immediately from the heated vial into a GC equipped with one of the two columns described in described in B.2.b under the following conditions:

   i. Injection temperature: 100° C.

   ii. Column temperature: 75° C.

   iii. FID temperature: 200° C.

   iv. Nitrogen carrier gas flow rate: 20 mL/min (stainless steel, 6′ × 1/8″ i.d. column) or 80 mL/min (glass, 6′ × 4 mm i.d. column).

   v. Hydrogen flow rate: 10 mL/min.

   vi. Compressed air flow rate: 100 mL/min.

   The best technique for injection is by "pumping" the syringe six times at moderate speed, filling the syringe slowly and making the injection within 30-90 seconds after the removal of the vial from the oven. Delay of injection results in drastically reduced response.

   Retention time for all lower boiling fractions is approximately 25 minutes. Do not perform next injection until these fractions are eluted.

f. Inject 10 μL each of the o, m, and p xylenes, and the ethyl benzene working standards.

DETERMINATIVE METHOD

F. ANALYTICAL QUANTITATION

Identification	Quantitation for gasoline is not possible because instrument response is not linear. Repeatability with the same tissue is good. Positive identification is made by comparing the retention times and relative peak heights of o, m, and p xylenes and ethyl benzene. The lower detection limit depends upon the sensitivity of the FID, but will be in the vicinity of 0.1 ppm gasoline.

DETERMINATIVE METHOD

H. HAZARD ANALYSIS

1. Method Title	Detection of Gasoline in Canned Meat by GC Headspace Analysis		
2. Required Protective Equipment	Safety glasses, heat-resistant gloves, lab coat.		
3. Procedure Steps		*Hazards*	*Recommended Safe Procedures*
	C. Reagents		
	Gasoline Petroleum ether Ortho xylene Meta xylene Para xylene Ethyl benzene	These reagents are very flammable and corrosive and the vapors are extremely irritating to the skin, eyes, and respiratory system.	These solvents should only be used in an efficient fume hood, away from any heat-generating devices.
4. Disposal Procedures	Organic solvents	See above	Segregate chlorinated from nonchlorinated and hold in designated storage cans until disposed of by the contractor or in-house specialist.

DETERMINATIVE METHOD

J. QUALITY ASSURANCE PLAN

1. Performance Standard

Because this is a semiquantitative procedure, normal performance standards do not apply.

Method sensitivity: ≥ 1 ppm.

2. Critical Control Points and Specifications

Because the analytical procedure is only for the detection of the presence of gasoline, there are no truly critical control points. However, there are specifications in technique.

Record	*Acceptable Control*
a. Vial sealing	First—aluminum foil; second—Teflon-coated rubber septum; third—aluminum cover cap.
b. GC injections	Inject immediately after completing step E.2.d. Use pumping technique described in written procedure. Do not perform next injection until all lower boiling fractions have eluted.

3. Readiness to Perform

a. Familiarization

i. Phase I: Standards—By at least 3 replicate injections of standard, show that instrument is functioning properly.

ii. Phase II: Minimum of three samples, with at least 2 positives.

Submit data on standards and samples to Chemistry Division for information.

NOTE: Phase I and Phase II may be performed concurrently.

iii. Phase III: Check samples for analyst accreditation.

(a) 14 samples submitted by supervisor.

(b) Report data to Chemistry Division, QSB.

b. Acceptability criteria.

No false positives or negatives.

NOTE: This section is only applicable if GAS is included in the regulatory monitoring sampling plan. For Emergency Response, a sufficient number of positive and negative controls are used to qualify the data set rather than the analyst.

DETERMINATIVE METHOD

J. QUALITY ASSURANCE PLAN (Continued)

4. Intralaboratory Check Samples

a. System, minimum contents.

i. Frequency: At least one check sample per analyst per set of samples.

ii. Random replicates chosen by supervisor or Laboratory QA Officer.

iii. Records to be maintained by analyst and reviewed by supervisor and Laboratory QC Officer for all replicate findings.

b. Acceptability criteria.

If unacceptable values are obtained, then:

i. Stop all official analyses by that analyst.

ii. Investigate and identify probable cause.

iii. Take corrective action.

iv. Repeat Phase III of section J.3 above if cause was analyst-related.

5. Sample Acceptability and Stability

a. Matrices: As required by exposure incident.

b. Sample receipt size: Varied; enough to accomodate all required testing.

c. Condition upon receipt: Frozen.

d. Sample storage:

i. Time: Maximum unknown; analyte dissipates from matrices.

ii. Condition: Frozen.