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TOLUENE,[a] BENZENE, XYLENE, AND OTHER VOLATILE COMPOUNDS ASSOCIATED WITH GLUE SNIFFING
Type B Procedure

Naresh C. Jain

APPARATUS

1. Centrifuge
2. Gas chromatograph (Varian Aerograph Hy-Fi Model 600-D), equipped with a flame ionization detector, a 0–1-mv recorder (Leeds and Northrup Speedomax H or a suitable substitute), and a spud stainless-steel column, 6' long and ¼" O.D., packed with 30% Carbowax 20M on acid-washed 60–80-mesh Chromosorb W

OPERATING CONDITIONS

Gas Flow Rates:
nitrogen – 35 ml/min
hydrogen – 28 ml/min
oxygen[b] – 100 ml/min
Column Temperature: 100°C or 130°C, depending upon the compounds to be separated (see Table 1 for retention times at these temperatures)
Injection Port Temperature: 160°C
Attenuation: X 32[c]

Condition the column overnight at 180°C with a flow of nitrogen carrier gas.

3. Shell vials, 2 dram, or suitable tubes, such as culture tubes
4. Blood pipettes, calibrated "to contain" (T.C.), 0.5 ml
5. Syringes (Hamilton 7101-N), 1 µl

REAGENTS

All chemicals used should be analytical reagent grade.

1. Sodium tungstate, 10 g/100 ml
   Dissolve 10 g of sodium tungstate in water and dilute to 100 ml.
2. Propyl acetate[d] internal-control solution, 20.9 mg/100 ml[e]

[a] Toluene has been selected to illustrate this method. Other volatile compounds can be similarly assayed.
[b] Oxygen is used for increased sensitivity, but air can also be used.
[c] This can be varied to give increased sensitivity.
[d] If propyl acetate is the volatile substance suspected, a different compound, such as acetone, should be used as internal control.
[e] It is not necessary to know the exact concentration of the internalcontrol solution as long as the same solution is used for both the known and unknown samples.

Pipet 0.25 ml of propyl acetate into a 1-liter volumetric flask and dilute to the mark with distilled water.

3. Toluene reference solution, 21.65 mg/100 ml
Pipet 0.25 ml of toluene into a 1-liter volumetric flask and dilute to the mark with distilled water. (If any of the other substances are suspected, prepare the appropriate reference solution).

SPECIMEN PREPARATION

1. If the specimen is blood or urine, no further preparation is needed.
2. If the specimen is stomach content, centrifuge it to obtain a workable volume (0.5 ml or more) of supernatant. Use the supematant as the test sample.
3. If the specimen is tissue, add 10.0 ml of the sodium tungstate solution to 5 g of tissue. Homogenize the tissue, then centrifuge the homogenate. Use the supernatant as the test sample.

PROCEDURE

Qualitative Determination

1. Examine the gas chromatograph to be sure that the operating conditions stated above are met. Refer to Table 1 for setting the column temperature (100°C or 130°C, depending upon the separation needed for the compounds under investigation).
2. Draw about 0.5 µl of distilled water[f] into the syringe, followed by about 0.5 µl of the test sample. Inject this mixture into the gas chromatograph.

TABLE 1.

RETENTION TIMES OF VARIOUS VOLATILE SUBSTANCES AT SPECIFIED GAS-CHROMATOGRAPHIC OPERATING CONDITIONS*

Compound	Column Temperature, 100°C		Column Temperature, 130°C	
	Retention Time, minutes	Relative Retention Time†	Retention Time, minutes	Relative Retention Time†
Acetaldehyde	2.56	0.73	1.25	0.60
Acetone	2.38	0.68	1.62	0.76
Benzene	4.31	1.23	2.81	1.33
Chloroform	5.62	1.61	3.25	1.53
Ethanol	3.50	1.00	2.12	1.00
Formaldehyde	1.81	0.52	1.38	0.65
Isobutanol	7.25	2.10	3.94	1.86
Isopropanol	3.19	0.91	2.06	0.97
Methanol	3.25	0.93	2.06	0.97
Methyl ethyl ketone	3.38	0.97	2.19	1.03
Propyl acetate	4.50	1.29	2.75	1.30
Toluene	6.94	1.98	4.06	1.92
Xylene	11.38	3.25	6.38	3.01

'Stainless-steel column, 6' x ¼", packed with 30% Carbowax 20M on Chromosorb W, AW, 60–80 mesh; nitrogen flow rate, 35 ml per minute: hydrogen flow rate, 28 ml per minute: oxygen flow rate, 100 ml per minute.
†To ethanol.

[f] This amount of water serves as a flushing agent; it avoids plugging of the syringe and helps keep it clean.

3. If no *peak* results from the injection, the test is negative. Make sure that the operating conditions are set for maximum sensitivity before reporting the result as negative. If there is a peak corresponding to the retention time of the compound suspected, perform the quantitative procedure to determine the amount present.

Quantitative Determination

1. Pipet 0.5 ml of the n-propyl acetate internal-control solution into each of two shell vials, one labeled "'known" and the other labeled "unknown".
2. Using a 0.5-ml T.C. blood pipette, transfer 0.5 ml of the toluene (or other appropriate) reference solution to the shell vial marked "known". Wash out the pipette three times with the internal-control solution in the shell vial to be certain that all of the known sample is transferred to the vial.
3. Using a second 0.5-ml T.C. blood pipette, transfer 0.5 ml of the unknown solution to the shell vial marked "unknown". Wash out the pipette three times with the internal-control solution in the sheli vial to be certain that the specimen is quantitatively transferred.
4. Inject duplicate 0.5-μl aliquots of the known and unknown solutions into the gas chromatograph, using 1-μl Hamilton syringes.
5. Measure the average peak heights corresponding to the substance to be quantitated and to the intemal-control solution for both the "known" and "unknown" samples.

CALCULATION

For blood, urine, or stomach content, when used directly!

$$C_x = C_S \times \frac{h_x}{h_S} \times \frac{R_S}{R_x}$$

and for tissue (*5* g) homogenized with 10.0 ml of the sodium tungstate solution,[g]

$$C_x = 3 \times C_S \times \frac{h_x}{h_S} \times \frac{R_S}{R_x}$$

where C_x = concentration of the specimen;
C_S = concentration of the reference solution for the volatile to be assayed;
h_x = peak height of the "unknown" mixture at the retention time of the substance to be assayed;
h_S = peak height of the substance to be assayed in the "known" solution;
R_S = peak height of the internal control in the "known" mixture;
R_x = *peak* height of the internal control in the "unknown" mixture.

INTERPRETATION (See p. 7.)

The presence of such solvents as benzene, methyl ethyl ketone, toluene, and xylene in blood and lungs is usually associated with glue sniffing. Most volatile compounds ingested and/or inhaled will be found unchanged in the biologic material. Isopropanol, however, is metabolized to acetone. If ingestion of

[g] If further dilutions are necessary because of high concentrations of volatiles present, make sure that thb is taken into account in the calculation.

isopropanol is suspected, test for both isopropanol and acetone. Acetone is also likely to be present in blood samples from diabetic patients.

ACCURACY AND PRECISION

The technique of injection is important and should be consistent for both the reference and unknown solutions. Best results are obtained when reference and unknown solutions have approximately the same concentrations, so that both give similar peak heights following injection of equal volumes. Also, for excellent accuracy and reproducibility, the ratios $\frac{h_S}{R_S}$ and $\frac{h_x}{R_x}$ should equal approximately 1.

REFERENCE

Jain, N. C., Direct Blood-Injection Method for Gas-Chromatographic Determination of Alcohols and Other Volatile Compounds. *Clin. Chem.*, *17*, 82,1971,