



STRYCHNINE

5016

C₂₁H₂₂N₂O₂

MW: 334.42

CAS: 57-24-9

RTECS: WL2275000

METHOD: 5016, Issue 3

EVALUATION: FULL

Issue 1: 15 May 1985

Issue 3: 26 February 2016

OSHA: 0.15 mg/m³

PROPERTIES: solid; MP 268 °C; VP not significant

NIOSH: 0.15 mg/m³/10h; Group I Pesticide

SYNONYMS: strychnidin-10-one

SAMPLING		MEASUREMENT	
SAMPLER:	FILTER (glass fiber, 37-mm)	TECHNIQUE:	HPLC, UV DETECTION
FLOW RATE:	1 - 3 L/min	ANALYTE:	strychnine
VOL-MIN:	70 L @ 0.15 mg/m ³	DESORPTION:	5 mL mobile phase
-MAX:	1000 L	INJECTION VOLUME:	20 µL
SHIPMENT:	routine	MOBILE PHASE:	aqueous 1-heptane sulfonic acid + CH ₃ CN; pH 3.5; 1 mL/min @ ambient temperature
SAMPLE STABILITY:	at least 7 days @ 25 °C [1]	COLUMN:	25 cm x 4.2-mm ID, packed with C18/US Pharmacopeia (USP) L1, 10-µm particle size
BLANKS:	2 to 10 field blanks per set	DETECTOR:	UV absorption @ 254 nm
ACCURACY		CALIBRATION:	solutions of strychnine in mobile phase
RANGE STUDIED:	0.073 to 0.34 mg/m ³ [3] 180-L samples)	RANGE:	10 to 70 µg per sample
BIAS:	4.4%	ESTIMATED LOD:	0.8 µg per sample [1,2]
OVERALL PRECISION ($\hat{S}_{r,T}$):	0.059 [1]	PRECISION (\hat{S}_r):	0.042 @ 13 to 62 µg per sample [1]
ACCURACY:	± 14.6%		

APPLICABILITY: The working range is 0.05 to 10 mg/m³ for a 200-L air sample.

INTERFERENCES: None known

OTHER METHODS: This revises Method S302 [2].

REAGENTS:

1. Strychnine, 98%.*
2. Acetonitrile, chromatographic quality.
3. Water, chromatographic quality, distilled.
4. Acetonitrile:water, 1:1 (v/v). Mix equal volumes of acetonitrile and distilled water.
5. Acetic acid, glacial.*
6. Acetic acid, 0.01 N. Dilute 0.6 mL glacial acetic acid to 1 L with distilled water.
7. 1-Heptanesulfonic acid sodium salt, 95%.
8. Calibration stock solution, 1 mg/mL strychnine in 0.01 N acetic acid. Use an ultrasonic bath to aid dissolution.
9. Mobile phase: Dissolve 1.1014 g 1-heptanesulfonic acid sodium salt in 980 mL 1:1 acetonitrile:water. Adjust pH to 3.5 with glacial acetic acid. Dilute to 1 L with 1:1 acetonitrile:water. Filter through 0.45- μ m PTFE filter and refrigerate. Prepare fresh weekly and if cloudiness appears.

*See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass fiber filter, 37-mm diameter, in cassette filter holder.
2. Personal sampling pump, 1 to 3 L/min, with flexible connecting tubing.
3. HPLC, UV absorption detector at 254 nm, recorder, integrator and column (page 5016-1).
4. Syringe filter, polypropylene housing, nylon membrane, 0.2- μ m pore size.
5. Syringe, 20- μ L.
6. Micropipets or syringes, 10- to 100- μ L.
7. Jars, glass, squat-form ointment, with polytetrafluoroethylene (PTFE)-lined screw caps.
8. Volumetric flasks, glass, 10-mL.
9. Pipet, 5-mL delivery, with pipet bulb.
10. Tweezers.
11. Ultrasonic bath.

SPECIAL PRECAUTIONS: Strychnine is a potent convulsant. Use impervious clothing, gloves and face shields when handling it [3,4]. Avoid skin contact with acetic acid as it may cause severe burns.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Sample at an accurately known flow rate between 1 and 3 L/min for a total sample size of 70 to 1000 L.

SAMPLE PREPARATION:

3. Transfer the filter carefully using tweezers to an ointment jar.
4. Add 5.0 mL mobile phase. Seal and gently swirl the jar to wet the filter.
5. Filter the sample solution through a syringe filter.

CALIBRATION AND QUALITY CONTROL:

6. Calibrate daily with at least six working standards over the range 1 to 70 μ g strychnine per sample (0.2 to 14 μ g/mL).
 - a. Add known amounts of calibration stock solution to mobile phase in 10-mL volumetric flasks and dilute to the mark.
 - b. Analyze together with the samples and blanks (steps 9, 10, and 11).
 - c. Prepare calibration graph (peak area vs. μ g strychnine).
7. Determine recovery (R) at least once for each lot of filters used for sampling in the calibration range. Prepare three filters at each of five levels plus three media blanks.
 - a. Deposit a known amount of calibration stock solution onto the filter. Allow filters to air dry.
 - b. Store samples overnight in ointment jars.
 - c. Prepare for analysis (steps 3 through 5) and analyze together with working standards (steps 9 through 11).

- d. Prepare a graph of R vs. μg strychnine recovered.
8. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration graph and R graph are in control.

MEASUREMENT:

9. Set HPLC system according to manufacturer's recommendations and to the conditions given on page 5016-1.
10. Inject sample aliquot using syringe, fixed volume sample loop or autosampler.
11. Measure peak area.

CALCULATIONS:

12. Determine the mass, μg (corrected for R) of strychnine found in the sample (W) and in the average media blank (B) from the calibration graph.
13. Calculate concentration, C, of strychnine in the air volume sampled, V (L):

$$C = \frac{W - B}{V}, \text{mg}/\text{m}^3$$

EVALUATION OF METHOD:

Method S302 was issued on February 17, 1978 [2], and evaluated over the range 0.073 to 0.34 mg/m^3 using a 180-L sample [1,5]. Overall precision, $\hat{S}_{r,T}$, was 0.059 with an average recovery of 100.9%, representing a non-significant bias. The concentration of strychnine was independently verified by analysis of filter samples with a UV spectrophotometer at 254 nm. Recovery was 0.98 in the range of 13.5 to 54.1 μg per sample. Collection efficiency was determined to be at least 98.8% for 180-L sample at a concentration to be 0.15 $\mu\text{g}/\text{mL}$. Average recovery for one-day old samples was 105% vs. 98.8% for seven-day old samples stored at room temperature. Since the filter samples for the recovery and stability studies were prepared with strychnine acetate rather than the free base, the recovery of the free base and the stability of such samples is unknown.

REFERENCES:

- [1] NIOSH [1976]. Back-up data report for Strychnine. In: Ten NIOSH analytical methods. Available as order No. PB288-629 from NTIS. Unpublished.
- [2] NIOSH [1979] Strychnine: Method S302. In: Taylor, DG ed. 2nd ed. (Vol 5). NIOSH manual of analytical methods. Cincinnati, OH: U.S. Department of Health, Education, and Welfare, Center for Disease Control, National Institute for Occupational Safety and Health, DHEW (NIOSH) Publication No. 79-141.
- [3] NIOSH [1978]. NIOSH criteria for a recommended standard: occupational exposure during the manufacture and formulation of pesticides. Cincinnati, OH: U.S. Department of Health, Education, and Welfare, Center for Disease Control, National Institute for Occupational Safety and Health, DHEW (NIOSH) Publication No. 78-174. Available as order No. PB81-227001 from NTIS.
- [4] NIOSH [1981]. Occupational health guidelines for chemical hazards. Cincinnati, OH: U.S. Department of Health and Human Services, Centers for Disease Control, National Institute for Occupational Safety and Health, DHHS (NIOSH) Publication No. 81-123.

- [5] NIOSH [1980]. Development and validation of methods for sampling and analysis of workplace and toxic substances. Cincinnati, OH: U.S. Department of Health and Human Services, Centers for Disease Control, National Institute for Occupational Safety and Health, DHHS (NIOSH) Publication No. 80-133.

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