

塩化アリル分析法

構造式	CH ₂ =CHCH ₂ Cl	分子量: 76.53	CASNo: 107-05-1
許容濃度等:	ACGIH 1ppm (TWA) 2ppm (STEL) (1ppm=3 mg/m ³)	物性等 融点 -135℃ 沸点 45℃ 蒸気圧 39.3kPa (20℃) 分配係数 log Pow = 1.5	
別名	アリルクロリド		
サンプリング		分析	
参照:【 NIOSH method No.1000】			
精度			
適用:			
妨害:			
参考文献: NIOSH method No.1000			

ALLYL CHLORIDE

1000



MW: 76.53

CAS: 107-05-1

RTECS: UC7350000

METHOD: 1000, Issue 2

EVALUATION: FULL

Issue 1: 15 February 1984

Issue 2: 15 August 1994

OSHA: 1 ppm
NIOSH: 1 ppm; STEL 2 ppm
ACGIH: 1 ppm; STEL 2 ppm
(1 ppm = 3.13 mg/m³ @ NTP)

PROPERTIES: liquid; d 0.938 g/mL @ 20 °C; BP 45 °C; MP
-135 °C; VP 39.3 kPa (295 mm Hg) @ 25 °C;
explosive range 3.3 to 11% v/v in air

SYNONYMS: 3-chloro-1-propene; 1-chloro-2-propene

SAMPLING		MEASUREMENT	
SAMPLER: SOLID SORBENT TUBE (coconut shell charcoal, 100 mg/50 mg) FLOW RATE: 0.01 to 1 L/min VOL-MIN: 16 L @ 1 ppm -MAX: 100 L SHIPMENT: routine SAMPLE STABILITY: at least 6 weeks @ 25 °C BLANKS: 2 to 10 field blanks per set		TECHNIQUE:	GAS CHROMATOGRAPHY, FID
		ANALYTE:	allyl chloride
		DESORPTION:	1 mL benzene; stand 30 min
		INJECTION VOLUME:	5 µL
		TEMPERATURE-INJECTION:	185 to 200 °C
ACCURACY RANGE STUDIED: 1.8 to 7.2 mg/m ³ [1] (100-L samples) BIAS: 6.4% OVERALL PRECISION ($\hat{S}_n$): 0.071 [1] ACCURACY: ±16.6%		-DETECTOR:	250 °C
		-COLUMN:	160 to 200 °C
		CARRIER GAS:	N ₂ , 30 mL/min
		COLUMN:	stainless steel, 1.2 m × 6-mm OD; 50/80 mesh Porapak Q or equivalent
		CALIBRATION:	standard solutions of allyl chloride in benzene
		RANGE:	0.05 to 1.5 mg per sample
		ESTIMATED LOD:	0.01 mg per sample
		PRECISION ($\bar{S}_j$):	0.023 [1]

APPLICABILITY: The working range is 0.16 to 3 ppm (0.5 to 10 mg/m³) for a 100-L air sample. The method is applicable to short-term samples taken at 1 L/min. The upper limit of loading depends on the concentrations of allyl chloride and other substances in the air, including water vapor.

INTERFERENCES: None identified.

OTHER METHODS: This is Method S116 in a revised format [2].

REAGENTS:

1. Benzene, chromatographic quality.*
2. Allyl chloride.
3. Hexane.
4. Calibration stock solution, 7.5 µg/µL. Dilute 75 mg allyl chloride (80.0 µL at 20 °C) to 10 mL with hexane.
5. Nitrogen, purified.
6. Hydrogen, prepurified.
7. Air.

*See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 6-mm OD, 4-mm ID, flame-sealed ends, containing two sections of activated (600 °C) coconut shell charcoal (front = 100 mg, back = 50 mg) separated by a 2-mm urethane foam plug. A silylated glass wool plug precedes the front section and a 3-mm urethane foam plug follows the back section. Pressure drop across the tube at 1 L/min airflow must be less than 3.4 kPa. Tubes are commercially available.
2. Personal sampling pump, 0.01 to 1 L/min, with flexible connecting tubing.
3. Gas chromatograph, flame ionization detector, integrator, and column (page 1000-1).
4. Vials, 2-mL, PTFE-lined caps.
5. Syringe, 10-µL, readable to 0.1 µL.
6. Volumetric flasks, 10-mL.

SPECIAL PRECAUTIONS: Benzene is a suspected human carcinogen. All work should be performed in a hood.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break the ends of the sampler immediately before sampling. Attach sampler to personal sampling pump with flexible tubing.
3. Sample at an accurately known flow rate between 0.01 and 1 L/min for a total sample size of 16 to 100 L.
4. Cap the samplers with plastic (not rubber) caps and pack securely for shipment.

SAMPLE PREPARATION:

5. Place the front and back sorbent sections of the sampler tube in separate vials. Discard the glass wool and foam plugs.
6. Add 1.0 mL benzene to each vial. Attach crimp cap to each vial.
7. Allow to stand 30 min with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

8. Calibrate daily with at least six working standards over the range 0.01 to 1.5 mg allyl chloride per sample.
 - a. Add known amounts of calibration stock solution to benzene in 10-mL volumetric flasks and dilute to the mark.
 - b. Analyze together with samples and blanks (steps 11 and 12).
 - c. Prepare calibration graph (peak area vs. mg allyl chloride).
9. Determine desorption efficiency (DE) at least once for each batch of charcoal used for sampling in the calibration range (step 8). Prepare three tubes at each of five levels plus three media blanks.
 - a. Remove and discard back sorbent section of a media blank sampler.
 - b. Inject a known amount of calibration stock solution directly onto front sorbent section with a microliter syringe.

- c. Cap the tube. Allow to stand overnight.
 - d. Desorb (steps 5 through 7) and analyze together with working standards (steps 11 and 12).
 - e. Prepare a graph of DE vs. mg allyl chloride recovered.
10. Analyze three quality control blind spikes and three analyst spikes to insure that the calibration graph and DE graph are in control.

MEASUREMENT:

11. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 1000-1. Inject sample aliquot manually using solvent flush technique or with autosampler.
NOTE: If peak area is above the linear range of the working standards, dilute with benzene, reanalyze and apply the appropriate dilution factor in calculations.
12. Measure peak area.

CALCULATIONS:

13. Determine the mass, mg (corrected for DE) of allyl chloride found in the sample front (W_f) and back (W_b) sorbent sections, and in the average media blank front (B_f) and back (B_b) sorbent sections.
NOTE: If $W_b > W_f/10$, report breakthrough and possible sample loss.

14. Calculate concentration, C , of allyl chloride in the air volume sampled, V (L):

$$C = \frac{(W_f + W_b - B_f - B_b) \times 10^3}{V}, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

Method S116 was issued on May 9, 1975 [2], and validated over the range 1.8 to 7.2 mg/m³ at 25 °C and 759 mm Hg using a 100-L sample [1]. Overall precision, $\hat{S}_{\text{Tr}}$, was 0.071 with average recovery 105.5%, representing a nonsignificant bias. The concentration of allyl chloride was independently verified by direct gas chromatography. Desorption efficiency was 0.943 in the range 0.15 mg to 0.6 mg allyl chloride per sample. Breakthrough (5% on back section) occurred between 210 and 240 min when sampling an atmosphere containing 7.56 mg/m³ allyl chloride at 0.94 L/min at 0% RH.

REFERENCES:

- [1] Documentation of the NIOSH Validation Tests, NIOSH, S116, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977).
- [2] NIOSH Manual of Analytical Methods, 2nd ed., V. 2, S116, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).

METHOD REVISED BY:

G. David Foley, NIOSH/DPSE; originally validated under NIOSH Contract CDC-99-74-45.