# Captan

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| Method number: | PV2093 |
| Target concentration: | 5 mg/m$^3$ ACGIH TLV. There is no OSHA PEL for captan. |
| Procedure: | Samples are collected by drawing known, volumes of air through OSHA versatile sampler (OVS-2) tubes, containing a glass fiber filter and two sections of XAD-2 adsorbent. Samples are extracted with acetonitrile and analyzed by high performance liquid chromatography (HPLC) using an ultraviolet (UV) detector. |
| Recommended air volume and sampling rate: | 60 minutes and 1.0 L/min (60 L) |
| Detection limit of the overall procedure | 0.02 mg/m$^3$ (based on the recommended air volume) |
| Status of method: | Partially validated method. This method has been only partially evaluated and is presented for information and trial use. |

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# 1 General Discussion

## 1.1 Background

### 1.1.1 History of Procedure

This evaluation was undertaken to determine the effectiveness of the OVS-2 sampling device for captan. It follows the procedure developed for carbaryl. (Ref. 5.1)

It should be noted that in this evaluation for captan several other analytes were also present in the analytical procedure. These other analytes are not mentioned in this evaluation, but can be seen on the sample chromatogram. (Sec. 3.5.2)

### 1.1.2 Toxic effects (This section is for information only and should not be taken as the basis of OSHA policy).

The acute oral $LD_{50}$ for rats is 12.5 ±3.5 g/kg, with death mostly occurring during the third and forth days. A National Cancer Institute carcinogenesis bioassay completed in 1977 was positive for mice. However, other bioassays for mice and rats have been negative. (Ref. 5.2)

There are indications that captan may cause skin irritation in some workers. (Ref. 5.2)

Based on the available toxicity data captan has been given a TLV of 5 mg/$m^3$ by the ACGIH. (Ref. 5.2)

### 1.1.3 Potential workplace exposure

No estimate of worker exposure to captan could be found. The estimated production of captan in 1972 was 17 million pounds. Captan is used as a protectant-eradicant fungicide. (Ref. 5.3)

### 1.1.4 Physical properties (Ref. 5.3-5.6)

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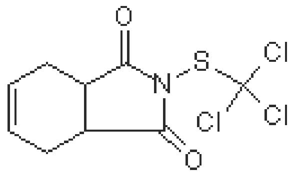
| Molecular weight: | 300.59 |
| Molecular formula: | $C_9H_8Cl_3NO_2S$ |
| CAS #: | 133-06-2 |
| Melting point: | 175 ºC |
| Appearance: | white, crystalline, odorless solid |
| Solubility | in g/100 mL of solvent at 25 ºC<br>acetonitrile 3.6<br>methanol 0.5<br>xylene 6.5 |
| Synonyms: | Captaf, Captex, Herpan, Orthocide, Pillarcap |
| Chemical name: | N-((trichloromethyl)thio)-4-cyclo-hexene-1, 2-dicarboximide. |
| Structure: |  |

## 1.2 Limit defining parameters

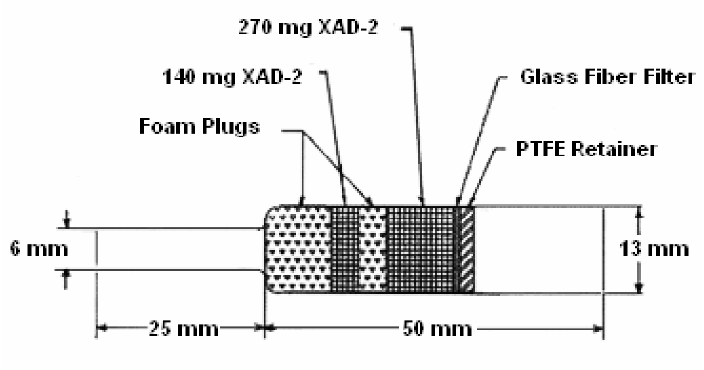
The detection limit of the analytical procedure is 6 ng per injection. This is the amount of analyte which will give a peak whose height is approximately five times the baseline noise.

## 2 Sampling procedure

### 2.1 Apparatus

2.1.1 Samples are collected by using a personal sampling pump that can be calibrated to within ±5% of the recommended flow rate with the sampling device in line.

2.1.2 Samples are collected with OVS-2 tubes, which are specially made 13-mm o.d. glass tubes that are tapered to 6-mm o.d.. These tubes are packed with a 140-mg backup section and a 270-mg sampling section of cleaned XAD-2. The backup section is retained by two foam plugs and the sampling section is between one foam plug and a 13-mm diameter glass fiber filter. The glass fiber filter is held next to the sampling section by a polytetrafluoroethylene (PTFE) retainer.



### 2.2 Reagents

No sampling reagents are required.

### 2.3 Sampling technique

2.3.1 Attach the small end of the OVS-2 sampling tube to the sampling pump with flexible, plastic tubing such that the large, front section of the sampling tube is exposed directly to the atmosphere. Do not place any tubing in front of the sampler. The sampler should be attached vertically (large end down) in the worker's breathing zone in such a manner that it does not impede work performance.

2.3.2 After sampling for the appropriate time, remove the sampling device and seal the tube with plastic end caps.

2.3.3 Wrap each sample end-to-end with a Form OSHA-21 seal.

2.3.4 With each set of samples, submit at least one blank. The blank should be handled the same as the other samples except that no air is drawn through it.

2.3.5 Bulk samples should be submitted for analysis in a separate container. Do not ship with the air samples.

### 2.4 Extraction efficiency

Two 13-mm glass fiber filters were each liquid spiked with 299.2 µg of captan. These two filters, along with a blank filter, were each placed in separate 4-mL vials which also contained 270 mg of XAD-2 adsorbent. These vials were stored overnight at room temperature, and then extracted with 2 mL of acetonitrile. The average extraction efficiency for these two filters (with the XAD-2 adsorbent present) was 90%.

### 2.5 Retention efficiency

Two OVS-2 tubes were each spiked with 299.2 µg of captan by liquid spiking the 13-mm glass fiber filter. Sixty liters of humid air were drawn through each tube. The two tubes were then extracted as in Section 3.4. No significant breakthrough to the backup section was observed. The average retention efficiency for these two tubes was 92%.

### 2.6 Sample storage

Two OVS-2 tubes were each spiked with 299.2 pg of captan as above. Sixty liters of humid air were drawn through each tube. These two tubes were stored for seven days at ambient temperature in a drawer. They were then extracted as in Section 3.4. No captan was recovered from the backup section of these tubes.

The average recovery after seven days of storage was 82%.

### 2.7 Recommended air volume and sampling rate

2.7.1 The recommended air volume is 60 L.

2.7.2 The recommended flow rate is 1.0 L/min.

### 2.8 Interferences

It is not known if any compounds will interfere with the collection of captan. Suspected interferences should be reported to the laboratory with submitted samples.

### 2.9 Safety precautions

2.9.1 Attach the sampling equipment in such a manner that it will not interfere with work performance or employee safety.

2.9.2 Follow all safety practices that apply to the work area being sampled.

## 3 Analytical procedure

### 3.1 Apparatus

3.1.1 A high-performance liquid chromatograph equipped with a UV detector, and manual or automatic injector. A Waters M6000A pump, Waters 710B autosampler, and Waters 440 UV detector with an extended wavelength module were used in this evaluation.

3.1.2 An HPLC column capable of separating captan from any interference. A 25-cm × 4.6-mm i.d. DuPont Zorbax ODS (6-µm) column was used in this evaluation.

3.1.3 An electronic integrator or other suitable means of measuring detector response. A Hewlett-Packard 3357 data system was used in this evaluation.

3.1.4 Vials, 4-mL glass with PTFE-lined septa.

3.1.5 Volumetric flasks, pipettes, and syringes for preparing standards, making dilutions, and performing injections.

### 3.2 Reagents

3.2.1 HPLC grade acetonitrile (ACN).

3.2.2 HPLC grade water. A Millipore Milli-Q system was used to prepare the water for this evaluation.

3.2.3 Captan, 99% pure (Chem Services Inc.).

3.3 Standard preparation

Stock standard solutions are prepared by adding acetonitrile to preweighed amounts of captan. Working range standard solutions are prepared by diluting stock solutions with acetonitrile. Stock and dilute standards are stored in a freezer.

3.4 Sample preparation

3.4.1 Transfer the 13-mm glass fiber filter and the 270-mg section of the sampling tube to a 4-mL glass vial. Place the first foam plug and the 140-mg section in a separate 4-mL glass vial. A small glass funnel can be used to facilitate the transfer of the adsorbent. Discard the rear foam plug. Do not discard the glass sampling tube; it can be reused after it has been cleaned with surfactant or suitable solvent.

3.4.2 Add 2.0 mL of acetonitrile to each vial.

3.4.3 Seal the vials with PTFE-lined septa and allow them to extract for one hour. The vials should be shaken by hand periodically during the one-hour extraction time.

3.5 Analysis

3.5.1 Instrument conditions

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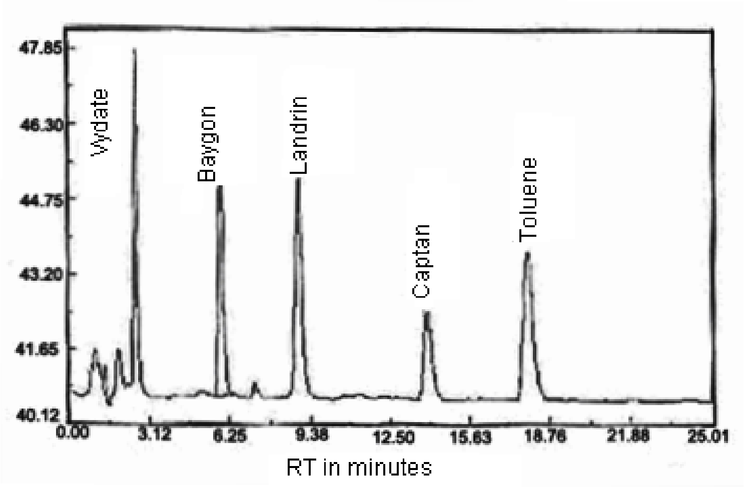
| Column: | 25-cm × 4.6-mm i.d. stainless steel column, packed with 6-µm DuPont Zorbax ODS |
| Mobile Phase: | 45% ACN / 55% water (v/v) |
| Flow rate: | 1 mL/min |
| UV detector: | 214 nm |
| Retention time: | 13.7 min |
| Injection volume: | 10 µL |

3.5.2 Chromatogram



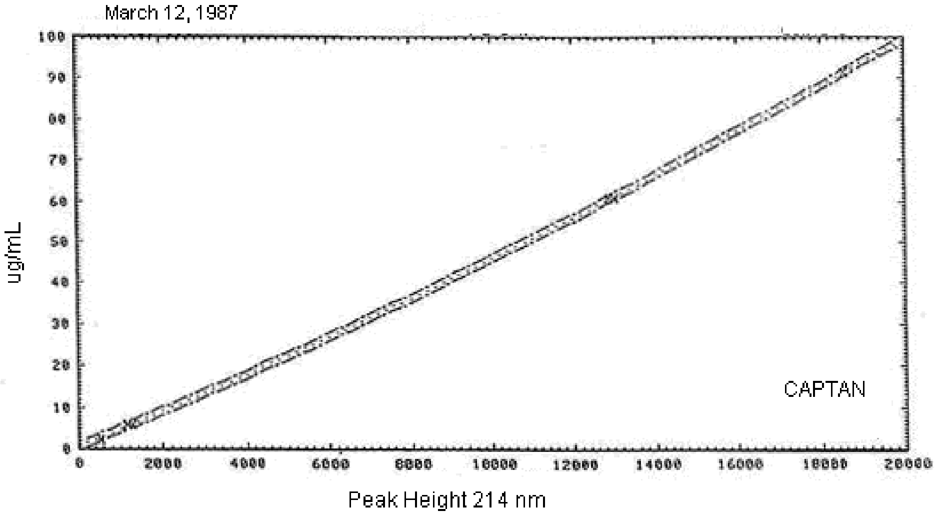
3.6 Interferences

3.6.1 Any compound having a similar retention time to the analyte is a potential interference. Generally, chromatographic conditions can be altered to separate interference from the analyte.

3.6.2 Retention time on a single column is not proof of chemical identity. Analysis by an alternate HPLC column, detection at another wavelength, comparison of absorbance response ratios, and confirmation by mass spectrometry are additional means of identification.

3.7 Calculations

3.7.1 A calibration curve is constructed by plotting detector response versus standard concentration.



3.7.2 The concentration of captan in a sample is determined from the calibration curve. If captan is found on the backup section, it is added to the amount found on the front section. Blank corrections for each section should be performed before adding the results together.

3.7.3 The air concentration is then determined by the following formula:

$$mg/m^3 = \frac{(\mu g/mL,\ blank\ corrected)(desorption\ volume,\ mL)}{(air\ volume,\ L)(desorption\ efficiency,\ decimal)}$$

3.8 Safety precautions

3.8.1 Avoid exposure to all standards.

3.8.2 Avoid exposure to all solvents.

3.8.3 Wear safety glasses, gloves, and lab coat in laboratory areas at all times.

## 4 Recommendations for further study

There appears to be some loss of captan with storage at ambient temperature. A more statistically valid storage study should be done to clarify this tendency. This method should be fully validated.

## 5 References

5.1 Burright, D., Method #63, "Carbaryl (Sevin)", OSHA Analytical Laboratory, unpublished, 1987.

5.2 "Documentation of the Threshold Limit Values and Biological Exposure Indices," American Conference of Governmental Industrial Hygienists Inc., fifth edition, 1986.

5.3 "Substitute Chemical Program, Initial Scientific and Mini economic Review of Captan", U.S. Environmental Protection Agency, Office of Pesticide Programs, Criteria and Evaluation Division, Washington, D.C., April 1975.

5.4 "Farm Chemicals Handbook," Meister Publishing Co., 1985.

5.5 Windholz, M., Ed. "Merck Index," 10th ed.; Merck and Co., Rahway, NJ, 1983.

5.6 "Chemical Information File", U.S. Department of Labor, Occupational Safety and Health Administration, Directorate of Science, Technology and Medicine, June 14, 1985.