Apron

Method no:	PV2102
Target Concentration:	5 mg/m^3 (arbitrary). There is no OSHA PEL or ACGIH TLV for Apron.
Procedure:	Samples are collected by drawing 60 L of air through glass fiber filters. Samples are extracted with acetonitrile and analyzed by high performance liquid chromatography (HPLC).
Recommended air volume and sampling rate:	60 minutes at 1 L/min (60 Liters)
Detection limit of the overall procedure based on the recommended air volume:	0.02 mg/m^3
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

The OSHA Analytical Laboratory received a set of samples for which an analysis of Apron was requested. The air samples had been collected on glass fiber filters at a flow rate of 1 L/min. This report describes the analytical method developed for Apron and a preliminary validation of glass fiber filter as the sampling medium.

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

The oral LD_{50} of Apron is 669 mg/kg in rats. The acute dermal LD_{50} is >3100 mg/kg for rats. It is relatively non-toxic to fish and wildlife. (Ref. 5.1)

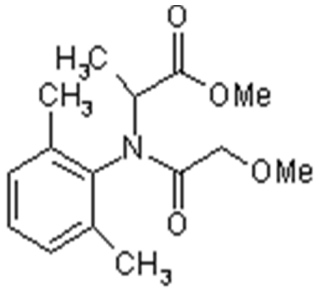
1.3 Potential workplace exposure (Ref. 5.3)

Apron is used as a fungicide in agricultural applications. No estimate of worker exposure during its production, formulation, and use as a fungicide could be found.

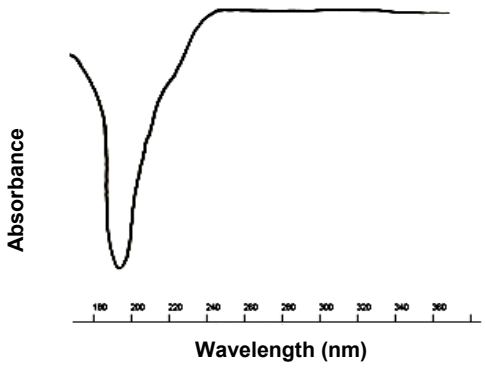
1.4 Physical properties (Ref. 5.1 and 5.2)

CAS no:	57837-19-1
Chemical name:	N-(2,6-Dimethylphenyl)-N-(methoxyacetyl) -DL-alanine methyl ester
Synonym:	Apron 2E; CG 117; CGA 48988; Metalaxil; Ridomil; Ridomil 2E; Subdue; Subdue 2E; Subdue 5SP; N-(Methoxyacetyl)-N-(2,6-xylyl)-DL-alanine methyl ester
Molecular formula:	$C_{15}H_{21}NO_4$
Molecular weight:	279.37
Appearance:	White crystals
Melting point:	71-72 °C
Vapor pressure:	2.2×10^{-6} mmHg at 20 °C
Solubility:	Solubility in water at 20 °C is 7.1 g/L. Apron is readily soluble in most organic solvents.

Structure:



UV Scan:



1.5 Detection limit of the analytical procedure

The detection limit of the analytical procedure is 4.3 ng per injection.

2 Sampling procedure

2.1 Apparatus and reagents

2.1.1 A personal sampling pump that can be calibrated to within ±5% of the recommended flow rate.

2.1.2 Glass fiber filter, 37-mm diameter, Gelman Type A, or equivalent.

2.1.3 Filter holder for 37-mm filters, Millipore M000037A0, or equivalent.

2.2 Sampling procedure

Use standard air sampling technique as specified in OSHA Instruction CPL 2-2.20A, Chapter II: Standard Methods for Sampling Air Contaminants.

2.3 Recommended air volume and sampling rate

2.3.1 The recommended air volume is 60 L.

2.3.2 The recommended sampling rate is 1 L/min.

2.4 Extraction efficiency

Three glass fiber filters were each spiked with ~16.35 µg of Apron. The filters were extracted with 5.0 mL of acetonitrile and analyzed. The average recovery of Apron was 100.4%.

Apron Extraction Efficiency		
YC1	16.07 µg	98.3%
YC2	16.60 µg	101.5%
YC3	16.56 µg	101.3%
average = 100.4%		

2.5 Retention efficiency

Three glass fiber filters were each spiked with ~16.35 µg of Apron. Humid air (50% RH, 183 L @ 1 L/min) was drawn through the filters. The filters were extracted with 5.0 mL of acetonitrile and analyzed. The average recovery of Apron was 97.9%.

Apron Retention Efficiency		
YC7	15.46 µg	94.6%
YC8	16.40 µg	100.3%
YC9	16.13 µg	98.7%
average = 97.9%		

2.6 Storage

Three glass fiber filters were each spiked with ~17.05 µg of Apron. Humid air (50% RH, 183 min @ 1 L/min) was drawn through the filters. The filters were stored at room temperature for 5 days, extracted with acetonitrile, and analyzed. The average recovery of Apron was 104.9%.

Apron Storage Recovery

YC10	17.05 µg	104.3%
YC11	17.05 µg	104.3%
YC12	17.33 µg	106.0^

average = 104.9%

2.7 Interferences

There are no known interferences to the sampling procedure.

3 Analytical method

3.1 Apparatus

3.1.1 High performance liquid chromatograph.

3.1.2 Alltech C18 column or equivalent.

3.1.3 UV detector.

3.1.4 Strip chart recorder.

3.2 Reagents

3.2.1 Water, HPLC grade.

3.2.2 Acetonitrile, HPLC grade.

3.2.3 Apron (Ridomil), EPA standard # F701.

3.3 Standard preparation

Weigh 2 to 4 mg of Apron in a 10-mL volumetric flask. Add acetonitrile to the mark. Dilute standards with acetonitrile to a suitable working range.

3.4 Sample preparation

Samples were extracted with 5.0 mL of acetonitrile and 30 minutes on a mechanical shaker.

3.5 Analysis

3.5.1 Instrument conditions

Column:	Alltech C18
Mobile phase:	60% acetonitrile/40% water
Detector:	198 nm (primary), 214 nm
Flow rate:	1.0 mL/min
Injection size:	25 µL
Retention time:	7.0 min

3.5.2 Chromatogram

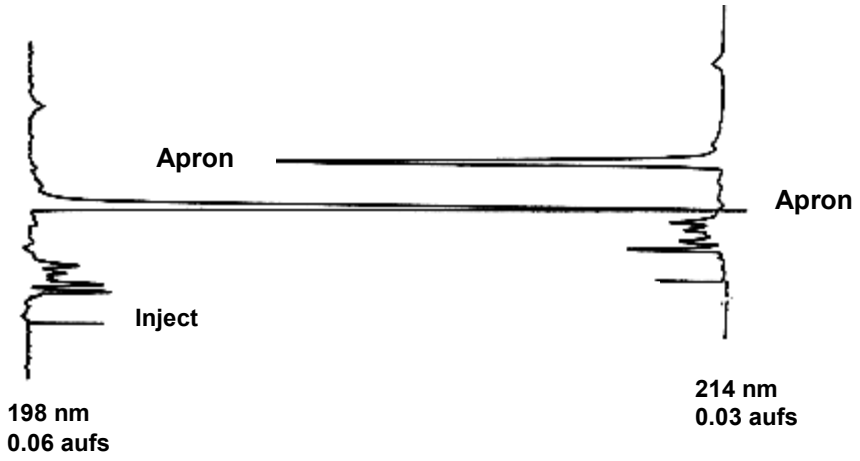


Figure 1. HPLC Chromatogram of Apron.

3.6 Interferences

3.6.1 Any collected compound that has the same retention time as Apron and absorbs at 198 and 214 nm is interference.

3.6.2 HPLC parameters may be varied to circumvent most interference.

3.6.3 Retention time alone is not proof of chemical identity. Confirmation by other means should be sought when possible.

3.7 Calculations

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

3.7.2 The concentration of a sample is determined from the calibration curve.

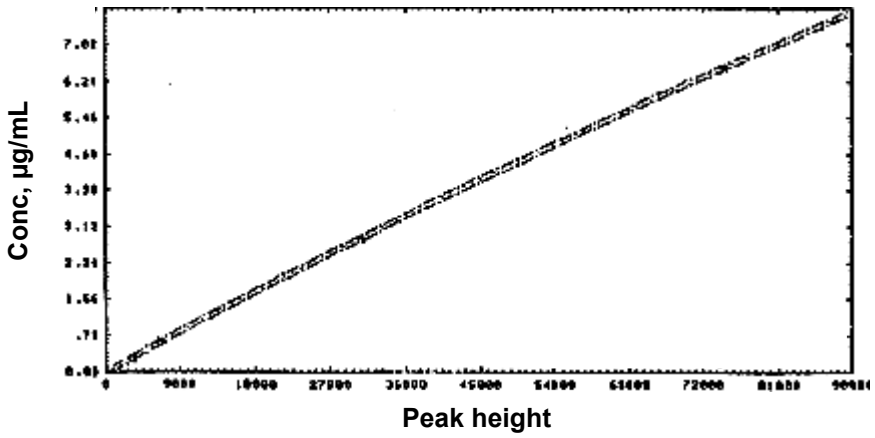


Figure 2. Apron calibration curve.

3.7.3 The air concentration is determined by the formula:

$$mg/m^3 = \frac{(\mu g/mL)(5mL)}{(air\ vol,\ L)(extraction\ efficiency,\ decimal)}$$

4 Recommendations for further study

The method should be fully validated.

5 References

5.1 Registry of Toxic Effects of Chemical Substances, 1983-84 Supplement, DHHS (NIOSH) Publication No. 86-103, Cincinnati, Ohio, 1986.

5.2 Merck Index, Tenth Edition, Merck & Co., Rahway, N.J., 1983.

5.3 Farm Chemicals Handbook, Meister Publishing, Willoughby, Ohio, 1981.