2-Aminopyridine
3-Aminopyridine
4-Aminopyridine

Method no.:	PV2143
Control no.:	T-PV2143-01-0605-M
Target concentration:	0.5 ppm (2-aminopyridine, 3-aminopyridine, and 4-aminopyridine)
OSHA PEL:	0.5 ppm (2 mg/m^3) (2-aminopyridine)
ACGIH TLV:	0.2 ppm (2-aminopyridine)
Procedure:	Samples are collected by drawing a known volume of air through 37-mm polystyrene cassettes containing two glass fiber filters coated with sulfuric acid separated by a spacer contained in a closed-face cassette. Samples are extracted with 3 mL of a solution of 0.1 N NaOH and analyzed by gas chromatography using a nitrogen-phosphorous detector (GC/NPD).
Recommended sampling time and sampling rate:	240 min at 1.0 L/min (240 L)
Reliable quantitation limit:	3.48 ppb (13.4 µg/m^3) 2-aminopyridine 5.23 ppb (20.2 µg/m^3) 3-aminopyridine 9.13 ppb (35.2 µg/m^3) 4-aminopyridine
Status of method:	Partially evaluated method. This method has been subjected to established evaluation procedures of the Methods Development Team and is presented for information and trial use.

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1. General Discussion

For problems with accessibility in using figures and illustrations in this method, please contact OSHA Salt Lake Technical Center at (801) 233-4900. These procedures were designed and tested for internal use by OSHA personnel. Mention of any company name or commercial product does not constitute endorsement by OSHA.

1.1 Background

1.1.1 History

Air samples collected using sulfuric acid coated glass fiber filters (GFF-H_2SO_4) were received at OSHA SLTC with requested analysis for aminopyridine. This partially-validated work was performed because SLTC had no sampling and analytical method for the three isomers of aminopyridine: 2-aminopyridine, 3-aminopyridine, and 4-aminopyridine. There are several OSHA methods that are used to collect aromatic amines using GFF-H_2SO_4: OSHA Methods 57, 65, 71, and 73.[1] These methods use extraction with an aqueous NaOH solution followed by either direct analysis, or by derivatization. The sensitivity for analysis of the basified extract analyzed by GC/NPD was sufficient at the target concentration of the aminopyridines, so no further derivatization was used.

The samples were extracted with 3 mL of 0.1 N NaOH with a mean extraction efficiency of 97.3, 97.7, and 97.5% for 2-aminopyridine, 3-aminopyridine, and 4-aminopyridine, respectively. The retention efficiency study showed no loss of 2-aminopyridine, 3-aminopyridine, or 4-aminopyridine from the front, spiked filter of a sampling train consisting of two cassettes connected in series, each cassette containing 2 GFF-H_2SO_4 separated by a spacer, that had 240 L of humid air drawn through them. The storage study showed little loss for samples stored for up to 15 days.

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

All three isomers of aminopyridine are toxic by ingestion, interaperitoneal, subcutaneous, and intravenous routes. Exposure to 2-aminopyridine can cause skin irritation, headache, dizziness, nausea, increased blood pressure, flushing of the extremities, with high exposures leading to convulsions and respiratory failure.[2] Exposure to 3-aminopyridine can cause eye, skin, and mucous membrane irritation, and nausea.[3] Exposure to 4-aminopyridine can cause eye, skin, and mucous membrane irritation, hallucinations and distorted perceptions, dyspnea, nausea or vomiting.[4]

1.1.3 Workplace exposure[5]

2-Aminopyridine is used in the synthesis of antihistamines and other pharmaceuticals. 3-Aminopyridine is used in the synthesis of drugs and dyes. 4-Aminopyridine is used as a drug and in synthesis of chemicals and other drugs.

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1 OSHA Methods 57, 65, 71, and 73, http://www.osha.gov/dts/sltc/methods/index.html (accessed 1/20/06).

2 *Documentation of the Threshold Limit Values for Chemical Substances,* 7th ed., American Conference of Governmental Industrial Hygienists Inc., Cincinnati, OH, 2001, vol. 1, 2-Aminopyridine 1-2.

3 3-Aminopyridine MSDS, www.sigmaaldrich.com/catalog/search/ProductDetail/ALDRICH/A78209 (accessed 2/15/06).

4 Lewis, R., *Hazardous Chemicals Desk Reference,* 4th ed., Van Nostrand Reinhold, New York, 1997, p 52.

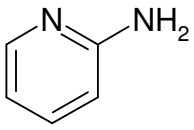
5 Lewis, R., *Hawley's Condensed Chemical Dictionary,* 14th ed., John Wiley & Sons Inc., New York, 2001, p 58.

1.1.4 Physical properties and other descriptive information

2-aminopyridine[2]

synonyms:	α-aminopyridine; *o*-aminopyridine; amino-2-pyridine; 2-AP; 2-pyridylamine; α-pyridylamine
IMIS[6]:	0165
CAS number:	504-29-0
boiling point:	210.6 °C (411 °F)
melting point:	58.1 °C (136.6 °F)
molecular weight:	94.12
flash point:	67.78 °C (154 °F) (closed cup)
appearance:	colorless to white solid
molecular formula:	$C_5H_6N_2$
odor:	characteristic unpleasant odor
solubility:	soluble in water, alcohol, benzene, and ether

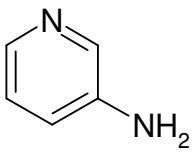
structural formula:



3-aminopyridine[4]

synonyms:	β-aminopyridine; 3-AP; 3-pyridylamine; *m*-aminopyridine; amino-3-pyridine; β-pyridylamine
IMIS[6]:	A174
CAS number:	462-08-8
boiling point:	251 °C (484 °F)
melting point:	64 °C (147 °F)
molecular weight:	94.12
flash point:	88 °C (190.4 °F) (closed cup)
appearance:	white to light yellow-brown crystals
molecular formula:	$C_5H_6N_2$
odor:	characteristic unpleasant odor
solubility:	soluble in water, alcohol, and ether

structural formula:

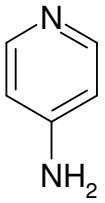


4-aminopyridine[4]

synonyms:	γ-aminopyridine; 4-AP; 4-pyridylamine; *p*-aminopyridine; amino-4-pyridine; γ-pyridylamine
IMIS[6]:	A173
CAS number:	504-24-5
boiling point:	273.5 °C (524.3 °F)
melting point:	158.9 °C (318 °F)
molecular weight:	94.12
appearance:	off-white to white crystals
molecular formula:	$C_5H_6N_2$

[6] OSHA Chemical Sampling Information. http://www.osha.gov/dts/chemicalsampling/toc/toc_chemsamp.html (accessed 2/15/06).

odor: characteristic unpleasant odor
solubility: soluble in water, slightly soluble in benzene, and ether
structural formula:



This method was evaluated according to the OSHA SLTC "Evaluation Guidelines for Air Sampling Methods Utilizing Chromatographic Analysis"[7]. The Guidelines define analytical parameters, specify required laboratory tests, statistical calculations and acceptance criteria. The analyte air concentrations throughout this method are based on the recommended sampling and analytical parameters.

1.2 Detection limit of the overall procedure (DLOP) and reliable quantitation limit (RQL)

The DLOP is measured as mass per sample and expressed as equivalent air concentration, based on the recommended sampling parameters. Ten samplers were spiked with equally descending increments of 2-aminopyridine, 3-aminopyridine, and 4-aminopyridine, such that the highest sampler loadings were 11.5 μg, 12.1 μg, and 22.8 μg, respectively. This is the amount spiked on a sampler that would produce a peak about 10 times the response for a sample blank. These spiked samplers were analyzed with the recommended analytical parameters, and the data obtained used to calculate the required parameters (standard error of estimate (SEE) and slope) for the calculation of the DLOP. The slope was 429.2 and the SEE was 137.8 for 2-aminopyridine. The slope was 495.8 and the SEE was 240 for 3-aminopyridine. The slope was 595.0 and the SEE was 502.7 for 4-aminopyridine. The RQL is considered the lower limit for precise quantitative measurements. It is determined from the regression line parameters obtained for the calculation of the DLOP, providing 75% to 125% of the analyte is recovered. The DLOP and RQL were 0.964 μg (1.04 ppb) and 3.21 μg (3.48 ppb), respectively for 2-aminopyridine, 1.45 μg (1.57 ppb) and 4.84 μg (5.23 ppb), respectively for 3-aminopyridine, and 2.53 μg (2.74 ppb) and 8.45 μg (9.13 ppb), respectively for 4-aminopyridine. The recovery at the RQL was 96.6% for 2-aminopyridine, 97.8% for 3-aminopyridine, and 95.4% for 4-aminopyridine.

[7] Evaluation Guidelines For Air Sampling Methods Utilizing Chromatographic Analysis, http://www.osha.gov/dts/sltc/methods/chromguide/index.html (accessed 2/15/06).

Table 1.2.1
Detection Limit of the Overall Procedure
for 2-Aminopyridine

mass per sample (μg)	area counts (μV•s)
0.00	0
1.15	323
2.30	637
3.45	1156
4.60	1944
5.75	2410
6.90	2964
8.05	3331
9.20	3645
10.4	4387
11.5	4745

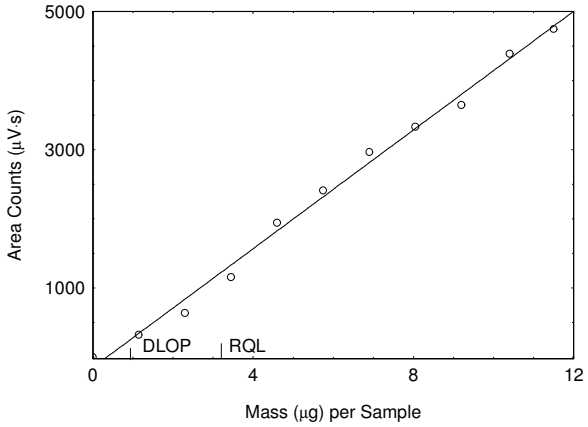


Figure 1.2.1 Plot of data to determine the DLOP/RQL for 2-aminopyridine. (y = 429x - 148; SEE = 137.8)

Table 1.2.2
Detection Limit of the Overall Procedure
for 3-Aminopyridine

mass per sample (μg)	area counts (μV•s)
0.00	0
1.21	494
2.42	1012
3.63	1424
4.84	2510
6.05	3326
7.26	3829
8.47	4293
9.68	4757
10.9	5123
12.1	5837

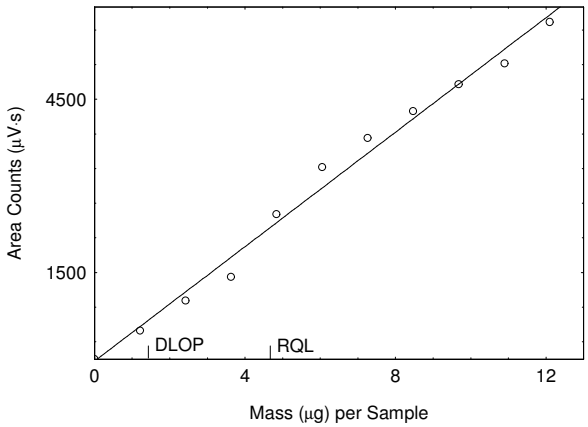


Figure 1.2.2 Plot of data to determine the DLOP/RQL for 3-aminopyridine. (y = 496x – 35.4; SEE = 240)

Table 1.2.3
Detection Limit of the Overall Procedure
for 4-Aminopyridine

mass per sample (μg)	area counts (μV•s)
0.00	0
2.28	503
4.56	1274
6.84	3338
9.12	4054
11.4	6634
13.7	7218
16.0	9036
18.2	9879
20.5	11102
22.8	13278

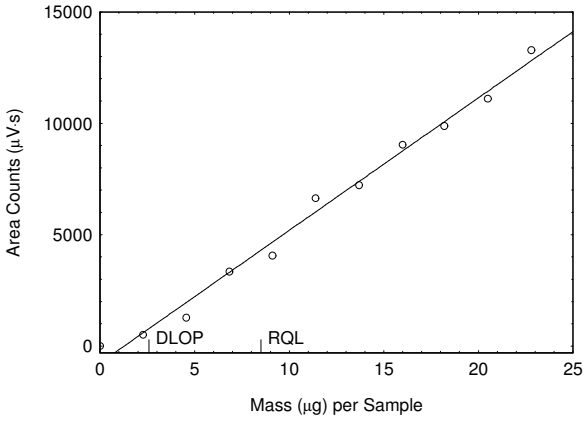


Figure 1.2.3 Plot of data to determine the DLOP/RQL for 4-aminopyridine. (y = 595x - 754; SEE = 502)

Below is a chromatogram of 2-aminopyridine, 3-aminopyridine, and 4-aminopyridine at the RQL. The recovery at the RQL was 96.6% for 2-aminopyridine, 97.8% for 3-aminopyridine, and 95.4% for 4-aminopyridine.

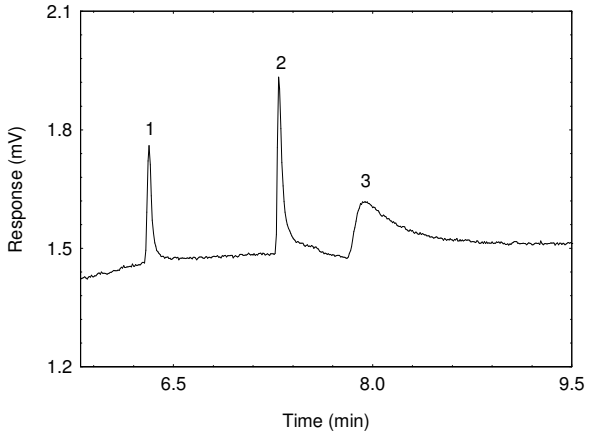


Figure 1.2.4 Chromatogram of the 2-aminopyridine, 3-aminopyridine, and 4-aminopyridine standard near the RQL. (Key: 1 = 2-aminopyridine, 2 = 3-aminopyridine, and 3 = 4-aminopyridine)

2. Sampling Procedure

All safety practices that apply to the work area being sampled should be followed. The sampling equipment should be attached to the worker in such a manner that it will not interfere with work performance or safety.

2.1 Apparatus

Samples are collected using a personal sampling pump calibrated, with the sampling device attached, to within ±5% of the recommended flow rate.

Samples are collected with 37-mm polystyrene cassettes containing two glass fiber filters coated with sulfuric acid, separated by a spacer. For this evaluation, commercially prepared sulfuric acid coated glass fiber filters in 37-mm polystyrene cassettes were purchased from SKC, Inc. (Catalog no. 225-9004, lot 3872).

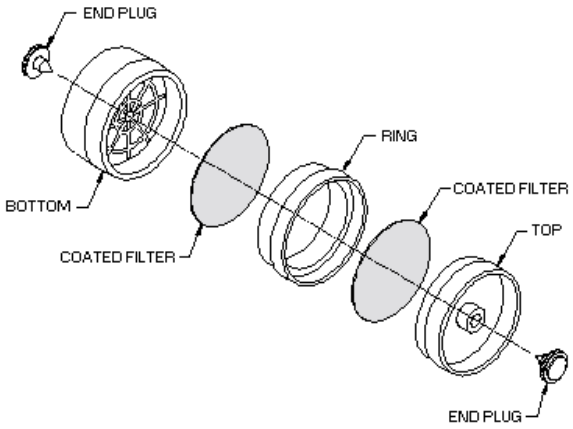


Figure 2.1 Expanded view of cassette assembly.

2.2 Reagents

None required

2.3 Technique

Immediately before sampling, remove the end plugs of the cassette. All cassettes should be from the same lot.

Attach the cassette to the sampling pump so that it is in an approximately vertical position with the inlet facing down during sampling near the worker's breathing zone. Position the sampling pump, cassette, and tubing so it does not impede work performance or safety.

Air being sampled should not pass through any hose or tubing before entering the cassette.

After sampling for the appropriate time, remove the sample, and replace the end plugs. Wrap each sample end-to-end with a Form OSHA-21 seal.

Submit at least one blank sample with each set of samples, making sure that it is from the same lot as the filters used for sampling. Handle the blank sampler in the same manner as the other samples except draw no air through it.

Record sample volumes (in liters of air) for each sample and any potential interferences.

Submit the samples to the laboratory for analysis as soon as possible after sampling. If delay is unavoidable, store the samples at refrigerator temperature. Ship any bulk samples separate from the air samples.

2.4 Extraction efficiency

The extraction efficiency was determined by spiking 16 GFF-H_2SO_4 with 2-aminopyridine, 3-amonopyridine, or 4-aminopyridine at 0.1 to 2 times the target concentration. These samples were stored overnight at ambient temperature and then extracted for 30 minutes using a lab shaker, and analyzed. The wet extraction efficiency was determined at 1 times the target concentration by spiking the analytes onto GFF-H_2SO_4 that had 240-L humid air (81% at 23 ℃) drawn through them. The mean extraction efficiency over the studied range was 97.3, 97.7, and 97.5% for 2-aminopyridine, 3-aminopyridine, and 4-aminopyridine, respectively. The wet recoveries were similar to the mean recoveries for all analytes.

Table 2.4.1
Extraction Efficiency (%) of 2-Aminopyridine

level		sample number						mean
× target concn	µg per sample	1	2	3	4	5	6	
0.1	46	97.8	99.1	97.9	98.2	96.0	95.2	97.4
0.25	115	96.3	97.8	99.3	95.2	98.1	94.8	96.9
0.5	230	98.0	98.2	98.6	97.1	96.8	97.3	97.7
1.0	460	98.0	96.5	97.2	95.9	96.7	96.8	96.9
1.5	690	95.9	95.0	97.7	98.4	98.3	98.2	97.3
2.0	920	98.3	98.2	99.2	95.4	96.2	97.1	97.4
1.0 (wet)	460	95.8	97.7	99.4	98.3	96.1	98.5	97.6

Table 2.4.2
Extraction Efficiency (%) of 3-Aminopyridine

level		sample number						mean
× target concn	µg per sample	1	2	3	4	5	6	
0.1	48	99.3	98.4	96.9	95.8	98.3	96.1	97.5
0.25	120	97.4	98.5	96.0	95.5	97.1	98.4	97.2
0.5	240	98.6	98.9	96.4	97.1	95.9	96.4	97.2
1.0	480	98.2	98.0	96.3	98.1	97.9	97.5	97.7
1.5	720	99.2	99.5	98.3	97.6	99.5	96.8	98.5
2.0	960	99.4	96.9	98.6	98.9	95.7	98.9	98.1
1.0 (wet)	480	96.7	95.1	99.3	97.9	98.6	98.4	97.7

Table 2.4.3
Extraction Efficiency (%) of 4-Aminopyridine

level		sample number						mean
× target concn	µg per sample	1	2	3	4	5	6	
0.1	49	98.0	96.9	95.3	95.3	98.4	97.6	96.9
0.25	123	95.8	98.2	96.3	97.9	96.6	98.0	97.1
0.5	245	98.8	97.7	96.5	99.0	99.0	96.2	97.9
1.0	490	95.6	99.3	96.3	99.6	96.8	97.3	97.5
1.5	735	95.2	97.4	97.7	99.7	98.9	97.4	97.5
2.0	980	99.2	95.5	99.5	99.4	98.0	97.0	98.1
1.0 (wet)	490	98.4	97.1	95.5	98.6	99.0	98.3	97.8

2.5 Retention efficiency

Six GFF-H_2SO_4 were spiked with 920 µg (1.00 ppm) of 2-aminopyridine, 960 µg (1.04 ppm) of 3-aminopyridine, and 980 µg (1.06 ppm) of 4-aminopyridine. These filters were placed in a 37-mm polystyrene cassette with a second unspiked GFF-H_2SO_4, with a spacer between them. This cassette was placed in a sampling train with a second cassette containing two unspiked GFF-H_2SO_4 separated by a spacer. These sampling trains had 240-L humid air (80% relative humidity at 23 ºC) pulled through them at 1 L/min. The samples were extracted and analyzed. The mean recovery was 98.0, 97.5, and 97.4% for 2-aminopyridine, 3-aminopyridine, and 4-aminopyridine, respectively. There was no analyte found on the back-up filter of the first cassette, or filters of the back-up cassette of any of the sampling trains.

Table 2.5.1
Retention Efficiency (%) of 2-Aminopyridine

section	sample number						mean
	1	2	3	4	5	6	
spiked GFF-H_2SO_4	98.2	96.5	98.1	97.6	98.7	99.0	98.0
rear GFF-H_2SO_4	0.0	0.0	0.0	0.0	0.0	0.0	0.0
back-up cassette	0.0	0.0	0.0	0.0	0.0	0.0	0.0
total	98.2	96.5	98.1	97.6	98.7	99.0	98.0

Table 2.5.2
Retention Efficiency (%) of 3-Aminopyridine

section	sample number						mean
	1	2	3	4	5	6	
spiked GFF-H_2SO_4	98.9	97.2	99.0	95.9	97.1	96.8	97.5
rear GFF-H_2SO_4	0.0	0.0	0.0	0.0	0.0	0.0	0.0
back-up cassette	0.0	0.0	0.0	0.0	0.0	0.0	0.0
total	98.9	97.2	99.0	95.9	97.1	96.8	97.5

Table 2.5.3
Retention Efficiency (%) of 4-Aminopyridine

section	sample number						mean
	1	2	3	4	5	6	
spiked GFF-H_2SO_4	98.5	95.3	96.7	98.1	96.8	99.2	97.4
rear GFF-H_2SO_4	0.0	0.0	0.0	0.0	0.0	0.0	0.0
back-up cassette	0.0	0.0	0.0	0.0	0.0	0.0	0.0
total	98.5	95.3	96.7	98.1	96.8	99.2	97.4

2.6 Sample storage

Fifteen GFF-H_2SO_4 were each spiked with 460 µg (0.50 ppm) of 2-aminopyridine, 480 µg (0.52 ppm) of 3-aminopyridine, and 490 µg (0.53 ppm) of 4-aminopyridine. These were assembled into 37-mm cassettes with a second unspiked GFF-H_2SO_4, with a spacer between the filters. These cassettes had 240 L of air (80% relative humidity at 23 ºC) drawn through them at 1 L/min. Three samples were analyzed immediately, and the rest were sealed. Six were stored at room temperature (23 ºC), while the other six were stored at refrigerated temperature (4 ºC). Three samples stored at room temperature and three samples stored at refrigerated temperature were analyzed after 8 days and the remaining six after 15 days. The amounts recovered indicate good storage stability for the time period studied.

Table 2.6.1
Storage Test for 2-Aminopyridine

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
0	97.2	98.9	96.9			
8	97.8	95.9	98.4	99.0	97.4	98.2
15	96.2	98.0	97.2	96.3	97.0	96.5

Table 2.6.2
Storage Test for 3-Aminopyridine

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
0	99.0	96.7	97.8			
8	98.5	97.1	96.2	97.8	98.1	97.0
15	95.7	95.3	93.6	95.4	96.3	98.1

Table 2.6.3
Storage Test for 4-Aminopyridine

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
0	98.4	98.9	95.7			
8	95.9	97.8	96.9	97.2	98.6	96.9
15	97.6	98.7	95.6	98.4	96.6	97.9

2.7 Recommended air volume and sampling rate

Based on the data collected in this evaluation, 240-L air samples should be collected at a sampling rate of 1 L/min for 240 minutes.

2.8 Interferences (sampling)

There are no known compounds which will severely interfere with the collection of 2-aminopyridine, 3-aminopyridine, or 4-aminopyridine.

Suspected interferences should be reported to the laboratory with submitted samples.

3. Analytical Procedure

Adhere to the rules set down in your Chemical Hygiene Plan. Avoid skin contact and inhalation of all chemicals and review all appropriate MSDSs.

3.1 Apparatus

A gas chromatograph equipped with a nitrogen-phosphorous detector. For this evaluation, an Agilent 6890 GC was used.

A GC column capable of separating 2-aminopyridine, 3-aminopyridine, and 4-aminopyridine from the extraction solvent and any potential interferences. A 60-m × 0.32-mm i.d. DB-1 (1.0-μm df) capillary column was used in this evaluation. Due to the caustic nature of the analytes and extraction solvent, a Siltek® injection port liner and a syringe with a PTFE (polytetrafluoroethylene) tipped plunger in the autosampler, were used in this evaluation. A syringe rinse of DI water was used in the autosampler.

An electronic integrator or some other suitable means of measuring peak areas. A Waters Empower2 Data System and an Agilent 3396 integrator were used in this evaluation.

Glass vials with PTFE-lined caps. For this evaluation 2-mL vials were used for the autosampler, and 4-mL vials used for sample extraction.

A dispenser capable of delivering 3.0 mL of extraction solvent to prepare standards and samples. If a dispenser is not available, a 3.0-mL volumetric pipet can be used.

A mechanical shaker. An Eberbach mechanical shaker was used in this evaluation.

Class A volumetric flasks, 10-mL and other convenient sizes for preparing standards.

Class A volumetric pipets and calibrated micropipets, for making analytical standards.

Micro-analytical balance capable of weighing to at least 0.01 mg. A Ohaus Galaxy 160D balance was used in this evaluation.

Optional: Centrifuge for spinning down the particles of the glass fiber filters in samples. An International Equipment Company Centra CL3 centrifuge was used in this method.

3.2 Reagents

2-Aminopyridine [CAS no. 504-29-0], reagent grade. Aldrich 99%+ lot 1852LI was used in this evaluation.

3-Aminopyridine [CAS no. 462-08-8], reagent grade. Aldrich 99% lot 11604CD was used in this evaluation.

4-Aminopyridine [CAS no. 504-24-5], reagent grade. Aldrich 98%+ lot 04609HR was used in this evaluation.

Sodium hydroxide [CAS no. 1310-73-2], reagent grade. Fisher 99%+ lot 046207 was used in this evaluation.

DI water, 18 MΩ-cm. A Barnstead NanoPure Diamond system was used to purify the water for this evaluation.

The extraction solvent solution was 0.1 N NaOH. This was prepared by placing 4 g of NaOH in a 1.0 liter flask and bringing it up to the mark with DI water.

3.3 Standard preparation

Prepare stock standards by weighing out known amounts of each aminopyridine into volumetric flasks and bringing it up to the mark with the extraction solvent. Dilutions of the stock standard are made with the extraction solvent to cover the range of 1 to 1000 µg/sample.

Bracket sample concentrations with standard concentrations. If, upon analysis, sample concentrations fall outside the range of prepared standards, prepare and analyze additional standards to confirm instrument response, or dilute high samples with extraction solvent and reanalyze the diluted samples.

3.4 Sample preparation

Remove the front and back GFF-H_2SO_4 from the cassette and carefully transfer each filter to a separate labeled 4-mL vial. Wipe the interior walls of the cassette with a GFF-H_2SO_4 wetted with a drop of DI water and place into a separate labeled 4-mL vial.

Add 3.0 mL of extraction solvent to each vial using the same dispenser as used for preparation of standards.

Immediately seal the vials with PTFE-lined caps, and shake the vials on a shaker for 30 minutes. Allow the vials to settle for 3 hours or spin them down on a centrifuge for 5 min at 2500 rpm. Transfer the clear supernatant to 2-mL autosampler vials for analysis.

3.5 Analysis

Gas chromatography conditions:
Zone temperatures:

column:	initial 110 °C, hold 1 min, program at 12 °C/min to 140 °C, then program at 20 °C/min to 200 °C, and hold 5 min
injector:	250 °C/min
detector:	260 °C

run time: 12 min
column gas flow: 1.5 mL/min (hydrogen)
injection size: 1.0 µL (10:1 split)
column: 60-m × 0.32-mm i.d. capillary DB-1 (df = 1 µm)

NPD conditions
hydrogen flow: 2 mL/min
air flow: 60 mL/min
nitrogen makeup flow: 10 mL/min

Peak areas are measured by an integrator or other suitable means.

Amine compounds tend to have carry-over of the previous injection onto the next injection. It may be necessary to do 3 injections/vial and throw out the first injection, which has the carry-over.

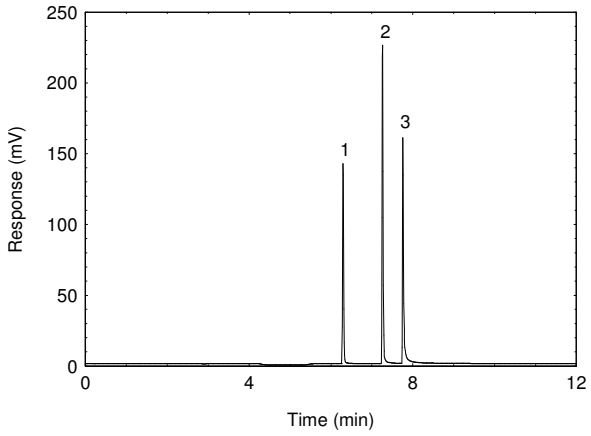


Figure 3.5.1 A chromatogram of 153 µg/mL 2-aminopyridine, 160 µg/mL 3-aminopyridine, and 163 µg/mL 4-aminopyridine. [Key: 1 = 2-aminopyridine, 2 = 3-aminopyridine, and 3 = 4-aminopyridine]

An external standard (ESTD) calibration method is used. A calibration curve can be constructed by plotting response of standard injections versus micrograms of analyte per sample (µg/mL x 3-mL sample volume = µg/sample). Bracket the samples with freshly prepared analytical standards over the range of concentrations.

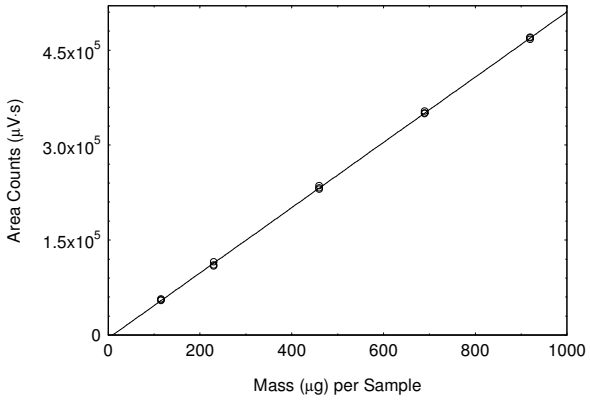


Figure 3.5.2 Calibration curve for 2-aminopyridine. (y = 515x – 5073)

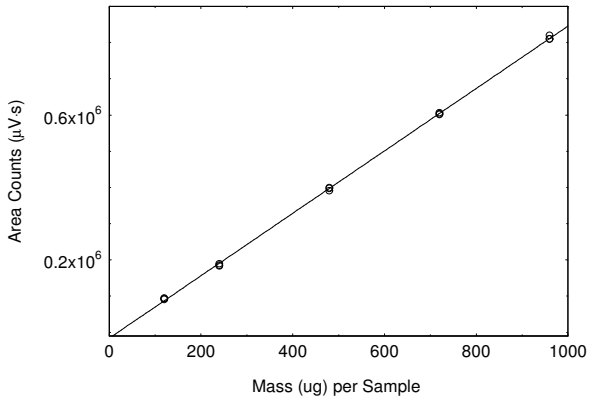


Figure 3.5.3 Calibration curve for 3-aminopyridine. (y = 862x – 1.62E4)

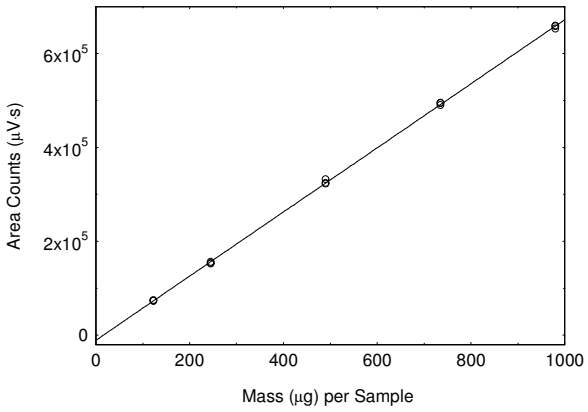


Figure 3.5.4 Calibration curve for 4-aminopyridine.
(y = 683x – 1.09E4)

The standard error of estimate was determined from the linear regression of data points from standards over a range that covers 0.25 to 2 times the TWA target concentration. Calibration curves were constructed and shown in Section 3.5 from the three injections each of five standards. The standard error of estimate are 14.6 µg/sample for 2-aminopyridine, 24.6 µg/sample for 3-aminopyridine, 21.0 µg/sample for 4-aminopyridine.

Table 3.5.1
Instrument Calibration for 2-Aminopyridine

standard concn (µg/sample)	x OSHA PEL	area counts (µV·s)		
115	0.25	56709	55129	54319
230	0.5	115267	110294	108679
460	1.0	230132	232084	235934
690	1.5	349728	350412	352502
920	2.0	466812	469129	470015

Table 3.5.2
Instrument Calibration for 3-Aminopyridine

standard concn (µg/sample)	x OSHA PEL	area counts (µV·s)		
120	0.25	92730	90937	94123
240	0.5	185430	183045	188932
480	1.0	397350	399268	390834
720	1.5	601368	603192	606149
960	2.0	809958	810234	820023

Table 3.5.3
Instrument Calibration for 4-Aminopyridine

standard concn (µg/sample)	x OSHA PEL	area counts (µV·s)		
123	0.25	72652	73349	74923
245	0.5	152286	155931	153423
490	1.0	322563	332012	324310
735	1.5	490098	494102	495341
980	2.0	659597	658042	653149

3.6 Interferences (analytical)

Any compound that produces a GC response and has a similar retention time as the analyte is a potential interference. If any potential interferences were reported, they should be considered before samples are extracted. Generally, chromatographic conditions can be altered to separate an interference from the analyte.

When necessary, the identity or purity of an analyte peak can be confirmed by GC-mass spectrometry.

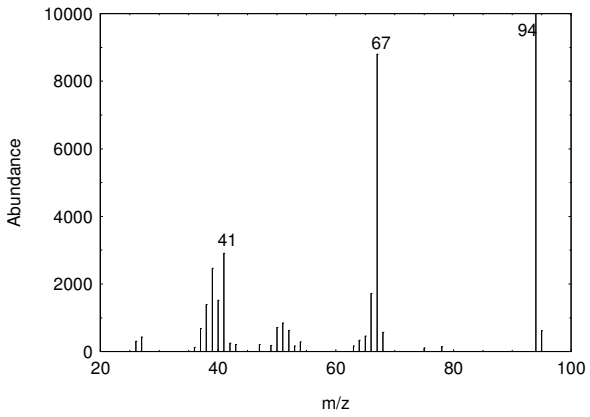


Figure 3.6.1 Mass spectrum of 2-aminopyridine.

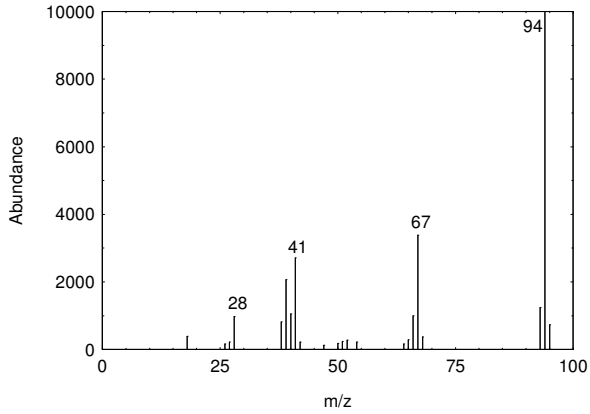


Figure 3.6.2 Mass spectrum of 3-aminopyridine.

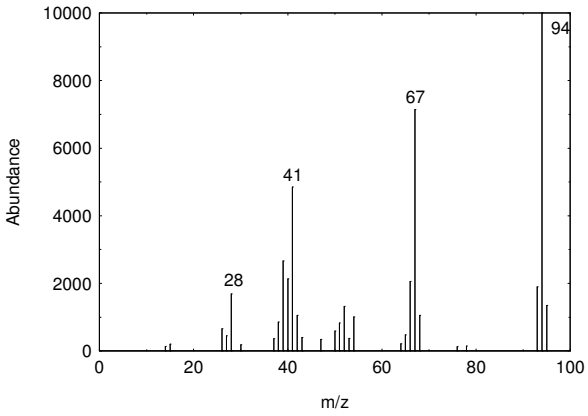


Figure 3.6.3 Mass spectrum of 4-aminopyridine.

3.7 Calculations

The amount of analyte per sampler is obtained from the appropriate calibration curve in terms of micrograms per sample, uncorrected for extraction efficiency. The results from each filter in the cassette are added together to calculate the total µg/sample. This amount is corrected by subtracting the amount (if any) found on the filters in the blank cassette. The blank-corrected results from the cassette wipe are included in the total µg/sample. The air concentration is calculated using the following formulas.

$$M = \left[\left(u_S - u_B\right) + \left(u_C - u_B\right)\right]$$

where u_S is ug/sample analyte in sample
u_C is ug/sample analyte in cassette wall wipe
u_B is ug/sample analyte in blank
M is microgram per sample

$$C_M = \frac{M}{VE_E}$$

where C_M is concentration by weight (mg/m^3)
M is micrograms per sample
V is liters of air sampled
E_E is extraction efficiency, in decimal form

$$C_V = \frac{V_M C_M}{M_r}$$

where C_V is concentration by volume (ppm)
V_M is molar volume at 25°C and 1 atm = 24.46
C_M is concentration by weight
M_r is molecular weight = 94.12

4. Recommendations for Further Study

Collection, reproducibility, and other detection limit studies need to be performed to make this a fully validated method.