

Arsenic Determination
Hydride Generation

Reagents

KBH₄ Solution 10%:

- dissolve 100g KBH₄, 1g KOH in H₂O
- dilute to 1L in a plastic 1L bottle

1N Iodine:

- dissolve 127g I₂, 256g KI in H₂O
- dilute to mark in 1L volumetric flask
- store in dark
- NOTE: use dry funnel to add I₂ to flask

N/50 Iodine:

- pipette 20ml 1N Iodine into 1L flask
- dilute to mark

Standards

a) Iodine Matrix Arsenic Standards

10ppm Arsenic:

- pipette 10ml @ 100ppm As into 100ml volumetric flask
- dilute to mark with N/50 Iodine

5ppm Arsenic:

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1ppm Arsenic:

- pipette 10ml @ 10ppm As into 100ml volumetric flask
- dilute to mark with N/50 Iodine

0.5ppm Arsenic:

- pipette 5ml @ 10ppm As into 100ml volumetric flask
- dilute to mark with N/50 Iodine

0.3ppm Arsenic:

- pipette 3ml @ 10ppm As into 100ml volumetric flask
- dilute to mark with N/50 Iodine

b) Water Matrix Arsenic Standards

5ppm Arsenic:

- pipette 5ml @ 100ppm As into 100ml volumetric flask
- add 5 ml HCL
- dilute to mark with distilled water

10ppm Arsenic:

- pipette 10ml @ 100ppm As into 100ml volumetric flask
- add 5ml HCL
- dilute to mark with distilled water

Arsenic Determination
Hydride Generation Method

1a) Digestion of Filters

- clean 50ml beakers with 5% HCL in water, on hot plate (reflux with watch glass in place) for one hour
- rinse with distilled water three times
- transfer the filter with acid washed forceps into clean beaker
- add 3ml concentrated Nitric Acid
- add 2ml concentrated Sulfuric Acid
- cover beaker with water glass, turn hot plate to three (low)
- the contents of the beaker are heated until a colourless gas appears, solution may have slight colour (digestion time is approximately three hours)

1b) Digestion of Swabs

- clean 250 ml Erlenmeyer flasks with acid water, rinse with distilled water
- transfer swab with acid washed forceps into flask
- rinse container that swab was in with concentrated Nitric Acid three times into flask
- add 10ml concentrated Sulfuric Acid
- add 10 ml concentrated Nitric Acid
- cover flask with watch glass
- digest on hot plate until swab is fully digested and dark acid fumes no longer exist

2a) Pump - Calibration

- fill four 250ml Erlenmeyer flasks to approximately 150ml with distilled water
- add 5ml concentrated HCL to each
- add stirring bars
- hook up four generation flasks to system
- check that pump is in following mode:

• Run	• Pump
	Dis
01	Dilute

- ensure that reagent lines are filled (no air pockets), this can be achieved by pressing and holding MAX mode for several seconds.

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2b) Calibration

- display should read 01
- press CAL, should read 0 (press X10 or -10 in order to achieve this)
- detach pump tubing from one generation flask and place in into a 50ml graduated cylinder
- press CAL
- press and hold firmly RUN key until 12ml of reagent has been dispensed (display reads RC during this period of time)
- press STOP
- pump has been calibrated to 12ml/hr
- reattach tubing to flask
- calibration is complete, calibration is only necessary on a daily basis

3) Run Preparation (for cleaning the generation system)

- if this immediately follows pump calibration the flasks attached to the system may be used

OR

- fill four 250ml Erlenmeyer flasks to approximately 150ml with distilled water
- add 5ml concentrated HCL to each
- add stirring bars
- attach to generation system
- place tubing ends in 1L beaker containing 5% acid-water
- dispense KBH₄ reagent at 120ml/hr for approximately 15 minutes prior to sample run

For 120ml/hr rate:

- pump has been calibrated at 12ml/hr (see calibration)
- press INCR (or DECR) until 01 is displayed
- press CAL, press X10 (so that 1 is displayed), press STOP
- press RUN
- run at this rate for 15 minutes approximately
- after 15 minutes, remove tubing ends from 1L beaker and rinse with distilled water
- NOTE: do not stop pump, let it continue
- reset timer for additional 5 minutes
- continue running at this rate for 5 minutes
- press STOP
- press MAX to clear lines of air pockets if necessary, before SAMPLE RUN

4. Sample Run

Preparation of Flasks **				Step 1	Step 2	Step 3	Corresponding 10 mls volumetric flasks * with 50 Iodine
Flask	Std/ Sample	Dist'led H2O	HCL	1.20 ml/h	120 ml/h	300 ml/h	
*1	3.0 mls @ 5 ppm	150 mls	5.0 mls	15 min	10 min	5 min	3 - 5
2	sample	150 mls	5.0 mls	15 min	10 min	5 min	3 - 5
3	sample	150 mls	5.0 mls	15 min	10 min	5 min	3 - 5
4	sample	150 mls approx	5.0 mls	15 min	10 min	5 min	3 - 5 approximately

*** Dedicate one flask to either "blank" or standard each run.

I.E.: 3.0 mls @ 5 ppm As standard

2.0 mls @ 10 ppm As standard

1.5 ppm (final Iodine solution)

2.0 ppm

4a) Flask Preparation

- wash four generation flasks ** with acid water, rinse three times with distilled water
- fill four 10ml volumetric flasks * with approximately 3 - 5ml Iodine (N/50) and clip to generation system
- transfer samples from beakers using not more than approximately 100ml of distilled water - be sure to wash down watch glass
- add standard to generation flask *** (pipette volumes as indicated above)
- fill flasks to approximately 150ml with distilled water
- add 5ml HCL and stirring bars
- hook up to hydride generation system
- NOTE: add 1ml of Antifoam to Erlenmeyer flask, to any sample which may contain organic matter (swabs)

4b) Water Samples

- pipette sample (required volume) or anticipated volume required
- 10ml, 50ml, 100ml
- add 5ml HCL to Erlenmeyer flask
- add stir bar

4c) Steps

In order to achieve desired rates of reagent addition:

i) 1.2ml/hr*

- press CAL, press -10 (display shows -1), press STOP
- display shows 01
- press RUN
- record correspondence between SAMPLE ID on Erlenmeyer flask and # on 10ml flasks
- run at this rate for 15 minutes
- press STOP

* i.e. $12 \times 10^{-1} = 1.2\text{ml/hr}$

ii) 120ml/hr**

- press DECR (display shows 1C)
- NOTE: 1C is equal to 100
- press RUN
- run at this rate for 10 minutes
- press STOP
- ** i.e. $1.2 \times 100 = 120\text{ml/hr}$

iii) 300ml/hr***

- press INCR (display shows 01)
- press CAL, press X10 (display shows 0), press STOP
- press INCR 25 consecutive times until 25 is displayed
- press RUN
- run at this rate for 5 minutes
- press STOP
- remove tubing from Iodine flasks immediately
- *** i.e. $12 \times 10^2 \times 25 = 300\text{ml/hr}$

4d) Cleanup and Analyze

- bulk volumetric flasks with N/50 Iodine to 10ml mark
- clean system with 100ml distilled water, 5ml HCL as in run preparation
- for end of day cleaning, fill a 1L beaker with acid water and submerge glassware and tubing
- analyze Iodine samples using direct flame AAS

STDS

0.5 ppm

1.0 ppm

2.0 ppm