

Sampling and Determination of Arsenic Trioxide in Baghouse Dust Shipments to Koppers.

A. Sampling Method

A two stage Ramsey / Galigher rotary sampling unit (Model 500 or equivalent) will be installed between the rotary vane feeder on the bottom of the loadout storage silo and the screw conveyor feeding the highway trucks (Figure 1).

The Ramsey / Galigher rotary sampler is a self contained dust tight unit designed to take sample cuts from a vertical stream of dry material. Sampling will be performed at predetermined intervals controlled by an automatic timer. The dust sample from the first rotary sampler will be screw conveyed to a secondary rotary sampler of similar design (Figure 2).

The dust sample from the second stage rotary sampling unit will fall by gravity into a Galigher Model 15C-200 Linear indexing sample collector (or equivalent). This unit will separate the final dust sample into four equal portions, each of which is representative of the consignment. The four equal portions are collected in plastic sample bags which will be sealed upon completion of the truck loading. Each of the four samples will be one to two pounds in size and labelled with a consignment number and date loaded.

B. Sample Handling

The four sealed plastic sample bags will be distributed as follows;

- One Sample to be shipped to Koppers with the loaded highway truck.
- One sample to be used by Giant Yellowknife Mines to determine the moisture content of the Arsenic Trioxide dust in that particular consignment.
- One sample to be used by Giant Yellowknife Mines to determine the concentration of arsenic, gold and iron in that particular consignment.
- One sample to be kept in reserve for a period of six months, in an impervious plastic container at Giant Yellowknife Mines, to be used by an umpiring agency in the event of a discrepancy as to the arsenic content of the consignment.

c. Determination of the Moisture content of Arsenic Trioxide Dust

Within 48 hours of truck loading, one sealed sample will be used to determine the moisture content of the truck consignment. The sample will be roughly split into two clean, tared metal weighing dishes. The wet dust weight of each sample will then be determined by weighing the samples on a top loading analytical balance with an accuracy rating of ± 0.5 grams. The balance will be standardized once per year by an approved balance servicemen.

The sample dishes will then be placed in a vacuum drying oven at an internal temperature of 120°F and left for a period

not less than 24 hours. The sample pans will then be reweighed on the same balance and the moisture content calculated as follows;

$$\text{Wet Dust weight} - \text{Dry Dust weight} = \text{weight of water}$$

$$\% \text{ Moisture} = \left(\frac{\text{weight of water}}{\text{wet dust weight}} \right) \times 100\%$$

The average of the two samples will be taken as the moisture content of that particular consignment.

D. Analytical Determination of the Arsenic, Gold and Iron content of each consignment.

A second sealed plastic sample bag will be chemically analyzed to determine the arsenic, gold and iron content^{of each consignment} using standard analytical techniques. These standard techniques are appended.

E. Determination of Total contained As_2O_3

The amount of dry As_2O_3 contained in each consignment will be calculated as follows;

Wet consignment weight : The weight of the fully loaded truck as measured at the N.W.T. Government's Enterprise truck scale minus the truck tare weight will be taken as the wet consignment weight.

Dry Consignment weight : The wet consignment weight

multiplied by $\left(1 - \frac{\% \text{ moisture}}{100}\right)$ will be taken as the dry consignment weight.

Concentration of Arsenic Trioxide :

The equivalent concentration of contained arsenic trioxide will be calculated by multiplying the concentration of ^{Total} arsenic determined analytically by 1.3203 .

The 1.3203 figure is determined as follows;

$$\frac{2 \times \text{Atomic wt. of AS} + 3 \times \text{Atomic wt. of O}}{2 \times \text{Atomic wt. of AS}}$$

where : Atomic wt. of AS = 74.9216

Atomic wt of O = 15.9994

The Total contained pounds of arsenic trioxide in each consignment will then be calculated as follows ;

Dry consignment weight (in pounds (advp.)) multiplied by the equivalent concentration of contained arsenic trioxide (in weight Percent / 100) .

1. Method for Analysis of commercial "Arsenic," Arsenious oxide, As_2O_3 .
from "Scott's Standard Methods of Chemical Analysis", Fifth Edition,
Volume 1, page 109, published by D. Van Nostrand Company
modified as follows:

Determination of Arsenic on Bag House Dust

Sample weight 0.2 grams.

Put sample in 600ml beaker and add 5-7 grams of NaOH plus 50ml water. ~~Allow dust~~ Allow the dust to dissolve.

Dilute to 200 ~~to~~ 300ml and add two drops of ^{METHYL} ~~Methyl~~ Orange

~~At~~ Acidify with HCl to color change.

Add $NaHCO_3$ to color change adding a 5gram excess.

Add 5ml of starch solution

Cool.

Titrate with standard Iodine to a purple end point.

When dissolving the bag house dust in the Concentrated NaOH there will be slight discoloration due to the presence of iron in the dust, but the main thing is to make sure that all the dust is dissolved before further dilution. It may be necessary to break down some of the ~~lump~~ lumps with a stirring rod.

It is also advisable to titrate a blank with the iodine as it does take a few drops of iodine to impart the proper depth of purple.

Standard Iodine Solution:

33.83 grams KI and 16.94 grams ^{I_2} ~~per~~ per liter

Dissolve the KI in water first, then pour the concentrated solution onto the iodine, as the iodine dissolves, transfer it into volumetric flask. Standardize against 0.2 grams of Arsenous Acid as in the normal assay. ~~Star~~ wht. arsenous acid $\times 0.7577$ = arsenic equivalent

Stable starch solution:

Measure out 1500mls of distilled water into 2000ml beaker. Take two 250ml beakers, add 12 grams of soluble starch to one and 40 grams of solid KOH to the other. Make a water slurry of the starch and dissolve the KOH in water. Pour the starch slurry into the 1500ml of water, then pour in the KOH solution slowly with stirring. Let the mixture stand for 4 hours or overnight. Add 55ml of HCl and adjust to pH 6, then add 12 mls of Acetic acid and dilute to 2000ml.

2. The concentration of gold and iron contained in samples of Baghouse dust will be determined using atomic absorption analytical techniques.

After dissolution of the baghouse dust, contained gold is extracted into a methyl isobutyl Ketone layer. The concentration of gold in this layer is measured using a Perkin Elmer model 303 Atomic absorption spectrophotometer.

The concentration of iron is measured on a Pye Unicam model SP 1900 Atomic absorption spectrophotometer. Both methods are standard however we can supply details upon request.