

TEST PROCEDURE AATM 112-01  
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## IODINE VALUE

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### DESCRIPTION OF METHOD

The iodine value method is reproducible only if the exact conditions of the test are carefully followed. Any changes in strength of reagent, sample size or reaction time may produce varying results.

The exact specified weight of the sample is accurately weighed into a glass stoppered iodine flask, and dissolved in chloroform. The measured volume of Hanus reagent is accurately added and after thorough mixing, is placed in the dark for exactly one hour. A corresponding reagent blank is simultaneously prepared.

At the end of the specified time, the reaction is stopped by adding potassium iodide and diluting with water to prevent loss of the free iodine. The amount of iodine present is determined by titrating with sodium thiosulfate using starch indicator. The difference between a reagent blank titration and the titration of the test sample represents the amount of iodine absorbed by the sample. The iodine value is calculated as grams of iodine per 100 grams of sample.

Hanus reagent is a solution of iodobromide in concentrated acetic acid. The iodine combines with double bonds slowly under these conditions. A large excess of the halogens must be present to complete the reaction. At the end of the reaction the unconsumed iodine should be greater than 60% of the total. If the sample titration is less than 60% of the blank titration, take a smaller sample and repeat the analysis.

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## **APPARATUS**

1. Iodine flasks, 250 ml. glass stoppered
2. 50 ml. Burette with a tolerance of  $\pm 0.07$  ml
3. 25 ml Class A volumetric pipette with a tolerance of  $\pm 0.07$  ml or appropriate dispenser
4. Dispensing device capable of dispensing 10 ml ( $\pm 1\%$ )
5. Dispensing device capable of dispensing 20 ml ( $\pm 1\%$ )
6. Graduated cylinder capable of containing 100 ml
7. Analytical balance capable of weighing to the nearest 0.1mg
8. Dispensing device capable of dispensing 3 ml

## **REAGENTS AND SOLUTIONS**

1. Chloroform, A.R.
2. Hanus Solution, A.O.A.C.
3. Potassium Iodide - 10% solution
4. Stabilized starch solution - Commercially prepared
5. Sodium thiosulfate, 0.1N - Commercially prepared or AATM 1004

## **PROCEDURE**

1. Accurately weigh the sample into a tared 250 ml iodine flask. Determine the sample size from the following table. Accurately record weight to the nearest mg.

<u>Anticipated Iodine Value</u>	<u>Sample Size</u>
0 - 30	0.8 $\pm$ .01 g
30 - 50	0.5 $\pm$ .01 g
50 - 100	0.25 $\pm$ .01 g
100 - 150	0.16 $\pm$ .01 g

2. Using the dispensing device, add 10 ml chloroform. Dissolve by swirling (NOTE: 50% aqueous products will not dissolve completely until Hanus solution is added).
3. Accurately add 25.0 ml Hanus solution and stopper immediately. Swirl to mix thoroughly and place in the dark.
4. Simultaneously prepare a corresponding reagent blank containing 10 ml chloroform and 25 ml Hanus solution. Stopper immediately and place the flask in the dark with the test sample.
5. After 60 minutes, using the dispensing device, add 20 ml 10% potassium iodide.
6. Add by graduated cylinder, 100 ml water rinsing the neck and side walls of the flask during addition of the water.

**PROCEDURE (cont.)**

7. Immediately titrate the solution using 0.1N sodium thiosulfate until the aqueous layer's color begins to lighten.
8. Using the dispensing device, add approximately 3 ml starch solution and continue titrating until the blue color of the aqueous layer begins to disappear.
9. Towards the end of the titration, at intervals, stopper the flask and shake vigorously to extract any iodine remaining in the chloroform layer.
10. When the end point appears to have been reached, again stopper and shake vigorously, allow the layers to separate and add two drops of starch solution to verify that no blue color is formed in the aqueous layer.
11. Read the burette accurately to two decimal places. Record.

**CALCULATIONS**

$$\text{Iodine Value} = \frac{(\text{ml blank} - \text{ml sample}) \times \text{N sodium thiosulfate} \times 12.69}{\text{g sample}}$$

**PRECISION**

<u>Product</u>	<u>Average</u>	<u>Std Dev</u>	<u>Coeff of Var</u>	<u>df</u>
Acetulan	7.42	0.107	1.448	26