MHSMP-76-5K

DETERMINATION OF CHLORIDE IN TATB

C. S. MacDougall

QUALITY DIVISION

OCTOBER - DECEMBER 1975

Normal Process Development
Endeavor No. 106

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SUMMARY

To determine total chloride content in TATB, the sample is burned in a Parr Bomb, and the chlorides formed are titrated potentiometrically using silver nitrate. The electrode system is a chloride specific ion sensing element and a single junction reference. Results on seven test samples agree generally within 10 percent with previous analyses by the Development Division. Replicate analyses show precision of the method to be ± 10 relative percent.

Inorganic chloride is assayed by dissolving the TATB in concentrated sulfuric acid. The HCl, which is liberated, is swept into dilute base by a nitrogen sweep and analyzed potentiometrically with the chloride specific electrode. E. Kohn has previously determined that organic chloride did not interfere in the measurement. Results on three test samples agree within 5 percent with Development Division analyses, with a ± 2 relative percent precision of analysis.

INTRODUCTION

One of the precursors of TATB, namely TCTNB, contains significant quantities of inorganic and organic chlorides. Consequently, the final TATB product must be analyzed for trace quantities of chloride. Kohn(*1*) has described a method for determining inorganic chlorides, while a classical combustion-titrimetric method is employed for total chloride. This paper concerns the transfer of the technology to the Quality Chemical Laboratory, and the slight modifications to the equipment.

EXPERIMENTAL

All potentiometric measurements were made with a Beckman Expandomatic pH meter. The electrodes were the Orion 94-17A chloride specific ion electrode and the Orion 90-01-00 single junction reference. A standard platinum-lined Parr Combustion bomb was used for conversion of organic chloride to measureable chloride. The arrangement of the analytical train for the inorganic chloride test is shown in Fig. 1.

DISCUSSION

TOTAL CHLORIDE IN TATB

A 0.25 g sample of TATB was combusted in a Parr bomb and the chloride formed was quantitatively transferred to a 150 mℓ Nalgene beaker. The chloride was then titrated potentiometrically with standardized $AgNO_3$. A first derivative curve was used to detect the endpoint. A group of

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(1) E. Kohn, Determination of Inorganic Chlorides in TATB, MHSMP-75-5K (October - December 1974).

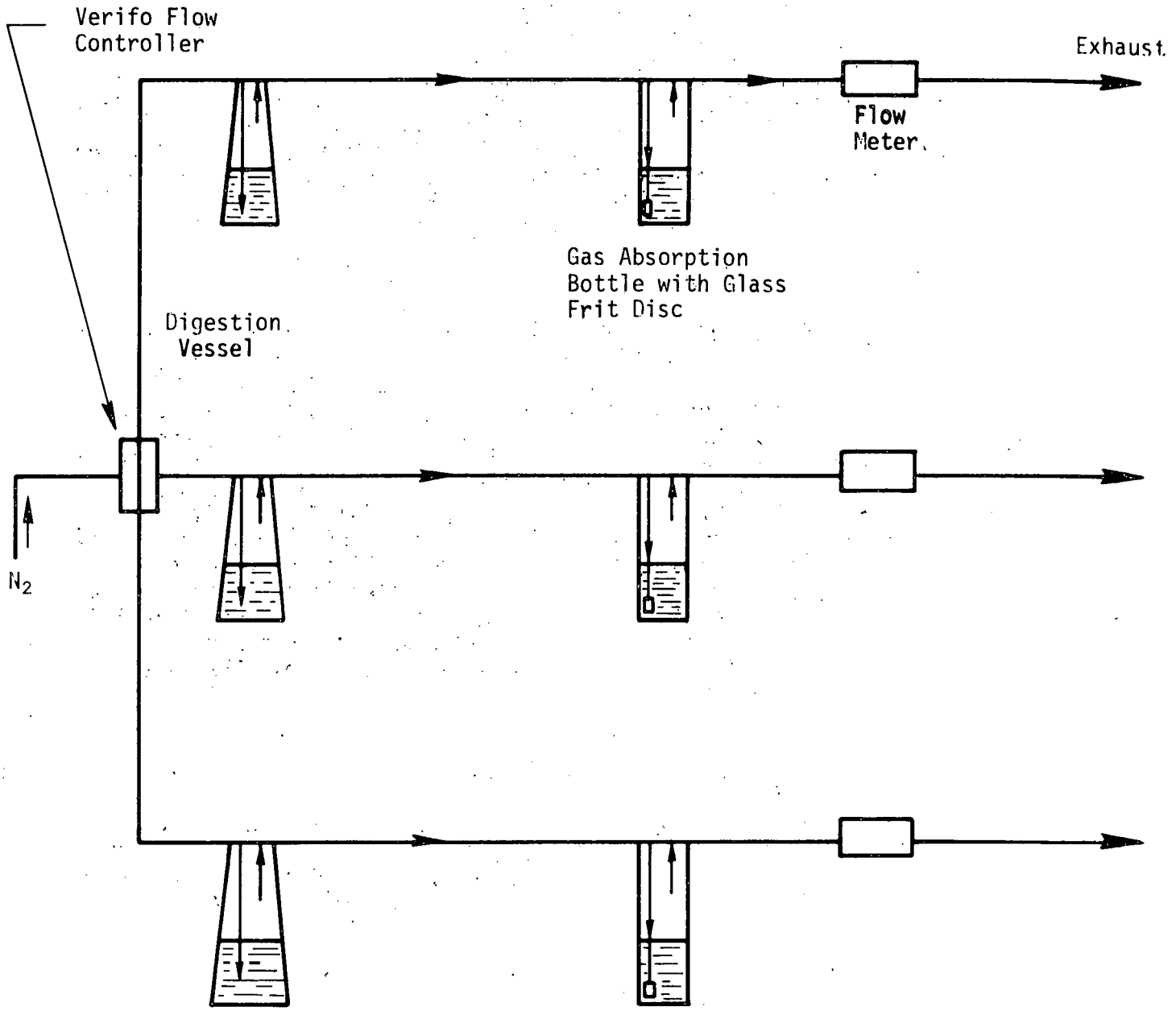

Fig. 1. Gas Train for Inorganic Chloride Analysis

seven samples, previously analyzed by Development Division, was assayed for total chloride. The only change in equipment was the electrode system. Table I shows the results of the analyses. As can be seen, our results (Table I, Column II) agree quite well with those measured in Development. In most cases agreement is within 12.5 percent. In order to determine precision of the method, one sample was analyzed repeatedly for total chloride (Table II). The relative standard deviation was about 10 percent. Most of the analyses compare within experimental error.

An attempt was made to directly measure chloride with the specific-ion electrode after combustion in the Parr bomb. Replication of readings on a standard chloride was within ± 2 percent utilizing a millivolt versus log concentration plot. However, the paraffin and decalin used for combustion in the Parr bomb interfered with the measurement. Therefore, further efforts along these lines were abandoned.

INORGANIC CHLORIDE IN TATB

A 5 g sample of TATB was weighed into a specially modified 50 mℓ Erlenmeyer flask. Forty mℓ concentrated H_2SO_4 were added to the Erlenmeyer, and all gas lines were quickly connected to the flask. Flow rates (150 mℓ/min) were checked before and after sample addition to ensure elimination of all gas leaks. The reaction proceeded for 2 hours, and all liberated HCl was collected in 50 mℓ 0.1 $\underline{N}$ NaOH. The collected chloride was then transferred to a Teflon beaker using 10 mℓ distilled H_2O as a wash solution. The chloride was titrated potentiometrically with 0.3 $\underline{N}$ $AgNO_3$, utilizing a first derivative curve for endpoint location. Three TATB samples previously analyzed by Development Division were assayed for inorganic chloride (Table III). Results determined by Development Division and Quality agree quite well, with the exception of 5198-16-01. This sample was of insufficient size for a standard 5 g assay, and represents only one analysis. Results still agree within 15%, even under adverse circumstances.

CONCLUSIONS

The chloride analyses used in Development Division have been successfully adapted to routine, production analyses of TATB. Modifications consisted mainly in a change of electrode systems and arrangement of the analytical train, and did not affect the accuracy of the assays.

ACKNOWLEDGEMENT

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Table I. Total Chloride in TATB

Sample	I Cl^- % Development	II Cl^- % Titration	III Cl^- % Direct Reading
5191-16-01	0.16	0.14	Inoperable
5174-16-02	1.24	1.40	
5195-16-02	0.43	0.36	
5223-16-01	1.79	1.80	
5238-16-02	0.74	0.79	
5239-16-02	0.51	0.61	
5240-16-01	0.66	0.71	

Table II. Precision of Measurement for Total Chloride in TATB

Run No.	Cl^- %
1	1.56
2	1.42
3	1.20
4	1.27
5	1.36
6	1.55
7	1.44
8	1.40
Total	1.40 ± 0.12

Table III. Inorganic Chloride in TATB

Sample	Inorganic Cl^- Development (%)	Inorganic Cl^- Quality (%)
5206-16-02	1.10	1.05 ± 0.02
5196-16-01	1.04	1.01
5198-16-01	1.32	1.21*

**One analysis only - insufficient sample*