ANALYSIS OF CHLORIDE IN TATB BY X-RAY FLUORESCENCE

C. C. Blair

DEVELOPMENT DIVISION

MASTER

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Mason & Hanger-Silas Mason Co., Inc.
Partex Plant
P. O. BOX 647
AMARILLO, TEXAS 79177
806-335-1581

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ABSTRACT

A procedure for the analysis of chlorides in TATB by X-ray fluorescence has been investigated. The instrument was calibrated using the method of standard additions. NaCl was used as the additive. Eighteen TATB samples were prepared and analyzed yielding a standard deviation of less than 0.004%.

INSTRUMENTATION

The equipment consists of a Philips Universal Vacuum X-ray Spectrograph with a chromium target X-ray tube, Germanium (III) analyzing crystal, gas flow detector, and solid state data measuring systems.

The chromium target X-ray tube was used because of its efficiency in exciting chlorine. The Germanium (III) crystal was used in order to separate the second order chromium line and its Compton scatter from the chlorine lines. The pulse height analyzer (PHA) was set to just pass the chlorine $K\alpha_1$ and $K\alpha_2$ lines. The $K\alpha_1$ line is observed at 92.24 degrees and the $K\alpha_2$ at 92.81 with the weight average occurring at 92.76. The spectrum was scanned from 90 to 94 in order to record a baseline on either end. The chlorine content was determined by using the height of the observed peak. A known standard was used during each run to help provide reproducible voltage and amperage settings. This provides space for three samples to be analyzed simultaneously.

EXPERIMENTAL

TATB Lot 5080-16-01 was used to prepare all standards since it contains a low chloride content (0.10% by the Parr Bomb method).

A standard solution of NaCl was prepared by dissolving 0.49545 ± 0.00005 g of NaCl in 100 ml of spectro grade water. Several 3.00000 ± 0.00005 g samples were weighed to mix with the NaCl solution. Standards were made by adding 1, 2, 3, 4, 5 and 6 ml of solution, respectively to the 3 g samples of TATB. For a 1 ml addition, the chloride content is $0.00100 \pm 0.00001\%$. The other standards were respectively, 0.1993%, 0.2985%, 0.3974%, 0.4959% and 0.5941%. The samples were stirred continually while the water evaporated.

Three samples were prepared at each percentage and two runs were made on each sample. The peak height of the reference was subtracted from the peak heights of the standards to find a height proportional to the chloride added. Table I lists those results.

The average peak height/0.1% Cl ratio for the thirty runs is 14.50 ± 0.43 . Peak height units are in 0.10-inch increments. This yields a standard deviation of less than 0.004% for the chloride analyses in the range of 0.1 to 0.7%. Table II compares X-ray with Parr Bomb analyses, duplicate analyses by X-ray, and X-ray analyses on conditioned material.

Table I

	<u>Lot No.</u>	<u>% Cl (Addition)</u>	<u>Peak Height</u>	<u>Peak Height (0.1% Cl)</u>
Run 1	5080-16-01 (17)	0.100	31.5	14.5
		0.100	32.0	15.0
		0.3974	72.0	13.8
		0.3974	71.5	13.7
		0.4959	86.0	13.9
		0.4959	86.0	13.9
Run 2	5080-16-01 (16)	0.2985	61	15
		0.2985	60	14.7
		0.3974	74	14.6
		0.3974	73	14.3
		0.5941	100+ ^a	
		0.5941	100+ ^a	
Run 3	5080-16-01 (15)	0.1993	45	15.0
		0.1993	45	15.0
		0.2985	58	14.4
		0.2985	58	14.4
		0.4959	85	14.1
		0.4959	85	14.1
Run 4	5080-16-01 (17)	0.1000	32	15.0
		0.1000	31.5	14.5
		0.3974	76	14.8
		0.3974	75	14.6
		0.4959	86	13.9
		0.4959	87	14.1
Run 5	5080-16-01 (16)	0.1000	31	15.0
		0.1000	31	15.0
		0.1993	46	15.0
		0.1993	46	15.0
		0.5941	100+ ^a	
		0.5941	100+ ^a	
Run 6	5080-16-01 (16)	0.1993	45	14.6
		0.1993	44	14.0
		0.5941	100+ ^a	
		0.5941	100+ ^a	
		0.2985	60	14.7
		0.2985	59	14.4

^aPeak height/0.1% Cl would be over 14 for these runs also.

Table II. Comparison of X-Ray with Parr Bomb Chloride Analysis

<u>Lot No.</u>	<u>X-Ray</u>	<u>Parr</u>
5131-16-01	0.506	0.47
5128-16-01	0.111	0.16
5127-16-01	0.565	0.53
5126-16-01	0.648	0.59
5120-16-01A	0.682	0.64
5120-16-01B	0.669	0.64
5121-16-01A	0.613	0.54
5121-16-01B	0.614	0.54
5122-16-01A	0.524	0.53
5122-16-01B	0.511	0.53
5125-16-01A	0.472	0.44
5125-16-01B	0.479	0.44
5142-16-01	0.586	0.51
5149-16-01	0.490	0.44
5150-16-01	0.75*	0.62
5153-16-01	0.85*	0.64
5120-16-01 - Ground	0.671	0.64
5120-16-01 - Ground	0.671	0.64
5120-16-01 - Ground	0.669	0.64

**Chloride percentages greater than 0.69% give greater than full scale deflection on the calibrated range.*

COMMENTS

From the difference observed in the peak heights of the Lot No. 5080-16-01 references and due to the difficulty in setting voltage and amperage, it is concluded that a reference must be used with every run. Further analysis of Table I shows that the reference material Lot No. 5080-16-01 contained $0.110 \pm 0.007\%$ chloride which agrees well with the 0.10 ± 0.02 obtained by the Parr Bomb method.

The X-ray analyses as a rule are higher than those from the Parr Bomb. Also, as the chloride content increases, the difference between X-ray and Parr Bomb results increases. It is suspected that at higher percentages of chloride the bomb results are erroneously low.

Samples taken directly from the same container show a variation in chloride content. Samples that were ground before being pelletized are equivalent, indicating inhomogeneity in the TATB.

It was observed that over a lengthy exposure to the vacuum and X-ray beam in the spectrometer, the chloride content decreased. This prompted regular changes of the reference.

For the data reported in this paper, the ratemeter setting was 1000 counts for full scale pen deflection. The most sensitive setting is 5 counts full scale. If the system is completely linear, 7 ppm chloride would give a deflection of 2 inches. This should be a realistic detection limit.

FUTURE WORK

The possibility of using peak area instead of height is being studied. The standard deviation may be significantly lowered by this means.