# Thermo. Titr. Application Note No. H-094

| **Title:** | **Determination of Boron in Ores by Fluoride Titration** |
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| ***Scope*:** | Determination of boron in ores of the element such as borax and ulexite. |

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| ***Principle*:** | Ores of boron such as borax and ulexite may be decomposed by acid digestion to yield boric acid. Acidic solutions of fluoride react exothermically with boric acid according to the equations: |

$$H_3BO_3 + 3H^+ + 3F^- \leftrightarrow HBF_3(OH) + 2H_2O$$

$$\underline{HBF_3(OH) + H^+ + F^- \leftrightarrow HBF_4 + H_2O} \qquad \text{(slow)}$$

$$H_3BO_3 + 4H^+ + 4F^- \leftrightarrow HBF_4 + 3H_2O$$

To form $HBF_4$, boric acid will decompose metal-fluoride complexes, and thus the determination of boric acid by fluoride is not affected by the presence of metals in solution. The titration of boric acid by fluoride also permits determination of boric acid independent of other acidic substances in the solution.

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| ***Reagents:*** | 2 mol/L potassium fluoride solution<br>Concentrated hydrochloric acid<br>25% v/v sulfuric acid solution |

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| ***Method:*** | *Basic Experimental Parameters:* |

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| Titrant delivery rate (mL/min.) | 5 |
| No. of endothermic endpoints | 1 |
| Data smoothing factor | 75 |
| Stirring speed | 12 |

*Procedure (for samples of ulexite in both powder and aqueous slurry form):*

*Ulexite Slurry.* Shake the slurry vigorously until all solids are suspended. Pour aproximately10g rapidly into a tared 250mL wide-necked Erlenmeyer flask and weigh accurately. Equip the flask with a magnetic stirrer follower and add 20mL concentrated HCl. Place a funnel in the neck of the flask. Bring the flask contents to the boil under stirring on a magnetic stirrer/hotplate, then add 100mL DI water. Bring the contents were to the boil again and hold

at the boil for 5 minutes. Remove the flask from the heat, and allow to cool. Transfer the contents quantitatively to a 250mL volumetric flask, made to the mark with DI water and mix well. Filter the contents through a dry Whatman no. 1 filter paper to remove precipitated silica acid, which might otherwise interfere.

*Ulexite powder.* Weigh 5g ulexite powder accurately into a tared wide-necked Erlenmeyer flask, and proceed as above.

*Titration:* For both types of samples, pipette a 15mL aliquot into a titration vessel, add 1.5mL of 25%v/v $H_2SO_4$, and add 20mL DI $H_2O$. A 15mL aliquot of solution prepared from ulexite slurry contained approximately 0.6g, and the same volume of solution prepared from the ulexite powder contained approximately 0.2g. *Note: If it is more convenient, other sample masses may be chosen to best suit the selection of volumetric glassware available in the laboratory. Aliquots should contain the approximate equivalent mass of sample used here*.

*Blank determination:*

A titration blank for the type of sample under examination is determined by titrating a range of aliquot sizes, and calculating the y-intercept (in mL) of a regression curve formed by plotting aliquot size (x-axis) against mL of titrant delivered (y-axis).

*Standardization:*

The KF titrant is standardized according to the procedure outlined in AN H-057

***Results:***

*(samples of ulexite supplied by a customer)*

| Sample | B % w/v |
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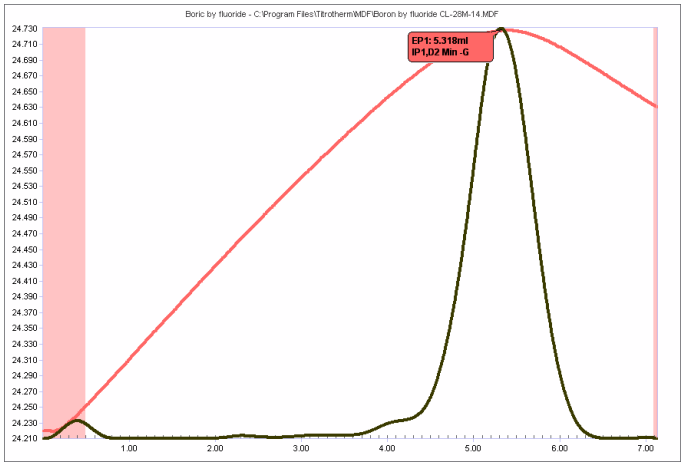
| Ulexite sample slurry #1 | 4.36±0.02 (n=6) |
| Ulexite sample slurry #2 | 4.34±0.03 (n=12) |
| Ulexite powder | 9.85±0.08 (n=7) |

***Calculation:***

$$\%B\ w/w = \frac{((\text{titre, mL} - \text{blank, mL}) \times \text{KF mol/L} \times 10.811 \times 100)}{(\text{sample vol, mL} \times 4 \times 1000)}$$

*(atomic mass of boron = 10.811)*

***Thermometric Titration Plot:***



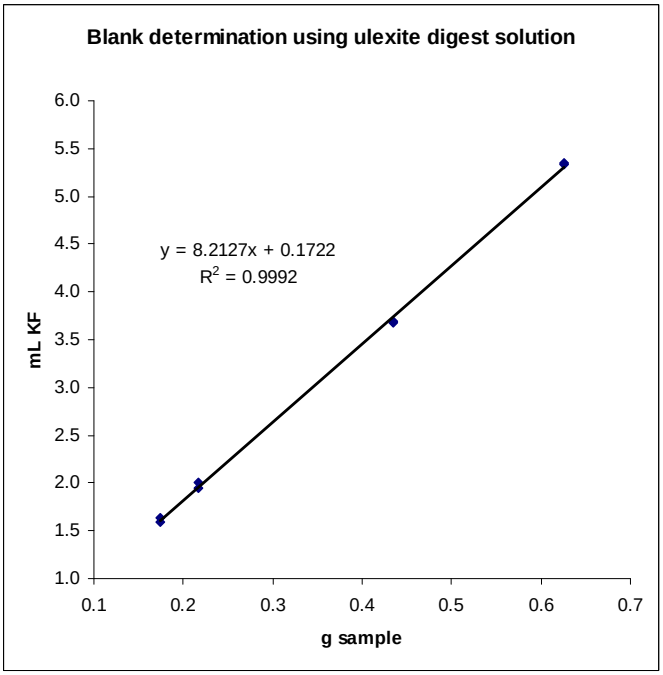

Legend:

Red = solution temperature curve

Black = second derivative curve

***Note:*** *the endpoint curvature is due to the relatively slow kinetics of the second reaction with fluoride. Nonetheless, good titration precisions can be obtained.*

***Blank determination:***




*Y-intercept = titration blank = 0.1722mL*