

SPECTROPHOTOMETRIC ANALYSIS 192

Determination of Metal Elements in Montmorillonite by AAS

Various ceramic materials are widely used in many fields and include oxides, silicates and their minerals as well as clay minerals. Montmorillonite is one of the clay minerals produced in mass in Japan in the form of the naturally occurring bentonite and it is widely used as an absorbent, catalyst and casting binder, among others. It is a feature of montmorillonite that its thermal property varies with the content of Fe and Mg. Here are shown examples of determination of Fe, Mg and Al, another principal component, in montmorillonite.

Sample Pretreatment

Weigh out 0.2g of sample into a platinum crucible, add 10 ml of hydrofluoric acid and 5 ml of perchloric acid, heat and decompose in the sand bath until the white fumes of perchloric acid are released, then allow to cool and add 10 ml of hydrochloric acid (~18%), boil, cool gradually, add purified water to make 50 ml, and use as the sample stock solution.

Analytical Conditions

The AA-680 atomic absorption spectrophotometer used in the analysis is capable of recording data in the form shown for Fe in Fig. 1. The analytical conditions for Fe, Mg and Al are shown in Table 1.

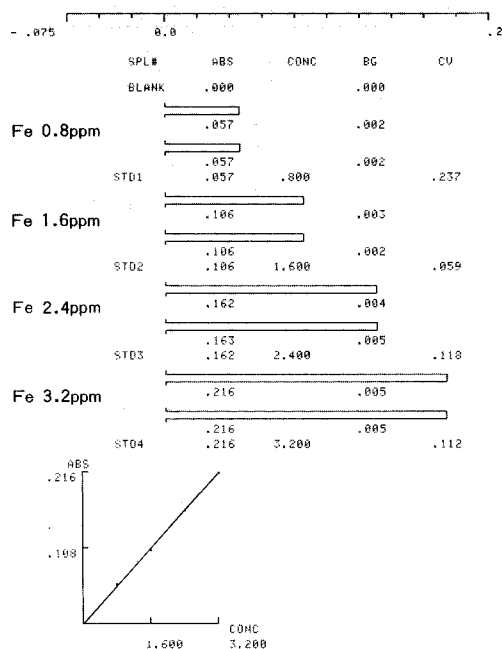
A final concentration calculation program was newly installed in the AA-680. This is a program for calculating and displaying the actual concentration of the element in the initial sample, by entering the sample weight W at the time of sample pretreatment, the specific volume L at the time of dilution, and the dilution factor M at the time of measurement. Calculated results for the elements are shown in Figs. 2, 4 and 6.

Table of Analytical Conditions

ELEMENT	ANALYTICAL LINE	FLAME	DILUTION OF SAMPLE
Fe	248.3 nm	Air-C ₂ H ₂	× 50
Mg	285.2 nm	Air-C ₂ H ₂	× 200
Al	309.3 nm	N ₂ O-C ₂ H ₂	× 25

In the determination of these elements, interference from other components was not detected, and all determinations were by the calibration curve method. However, in the measurement for magnesium, 2000 ppm of lanthanum was added to both the standard and sample solutions.

Determination of Fe



C=M A÷2+N A
M= 7.254E-01
N= 1.462E+01
S= 2.890E-04

STD DATA OMIT ?
1. OMIT: EOMIT3 EDATA NO.3 EEE EEE
2. NOT OMIT: EEE EEE
CAL. CURVE READY

BLANK	.000		.004	
	.102		.001	
Sample A (×50)	.101		.001	.924
	.102	1.506		
	.155		.002	
Sample B (×50)	.156		.002	.320
	.156	2.298		

**** DATA LIST ****
SPL# DATA SPL# DATA SPL# DATA SPL# DATA
101 1.506 102 2.298

DATE() OPERATOR()

Fig. 1 Measurement for Fe

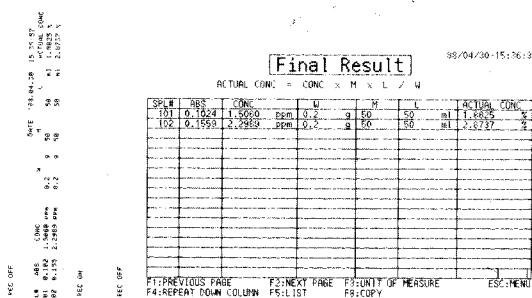
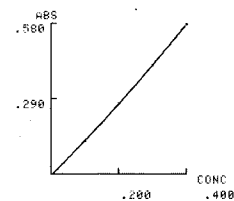
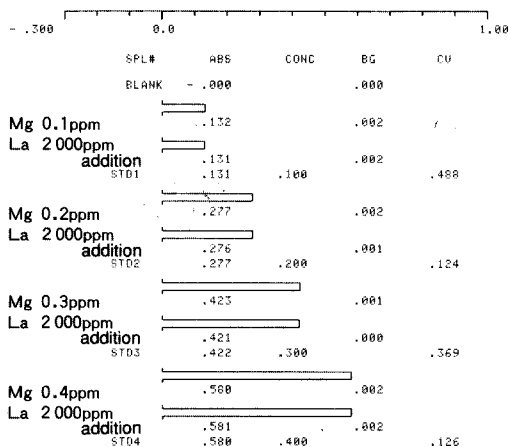


Fig. 2 Final Result for Fe

Determination of Mg



C=H A=2+H A
M=1.282E+01
H=7.639E+01
S=3.854E-05

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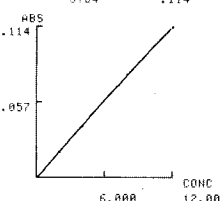
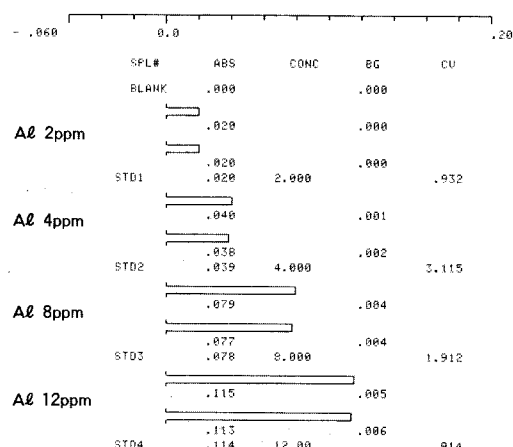
SPL#	DATA	SPL#	DATA	SPL#	DATA	SPL#	DATA
BLANK	.003		.000				
La 2000ppm addition	.393		.004				
Sample A (X200)	.395	.280	.005	.303			
La 2000ppm addition	.384		.005				
Sample B (X200)	.363	.260	.005	.222			

**** DATA LIST ****
SPL# DATA SPL# DATA SPL# DATA SPL# DATA
101 .280 102 .260

DATE(. .) OPERATOR()

Fig. 3 Measurement of Mg

Determination of Al



C=H A=2+H A
M=5.822E+01
H=9.788E+01
S=3.273E-05

STD DATA OMIT ?
1. OMIT: [OMIT] [DATA NO.] [EE] [EE]
2. NOT OMIT: [EE] [EE]
CAL. CURVE READY

SPL#	DATA	SPL#	DATA	SPL#	DATA	SPL#	DATA
BLANK	.003		.012				
Sample A (X25)	.046	4.622	.002	1.896			
Sample B (X25)	.063	6.487	.004	1.870			

**** DATA LIST ****
SPL# DATA SPL# DATA SPL# DATA SPL# DATA
101 4.622 102 6.487

DATE(. .) OPERATOR()

Fig. 5 Measurement of Al

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Final Result

ACTUAL CONC = CONC * M * L / W

SPL#	ABS	CONC	W	M	L	ACTUAL CONC
101	0.394	0.280	0.2	200	50	11.454
102	0.363	0.260	0.2	200	50	10.927

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Fig. 4 Final Result for Mg

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Final Result

ACTUAL CONC = CONC * M * L / W

SPL#	ABS	CONC	W	M	L	ACTUAL CONC
101	0.046	4.622	0.2	25	50	1.894
102	0.063	6.487	0.2	25	50	2.524

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Fig. 6 Final Result for Mg



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