

X-Ray (XRF) 가

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Evaluation of Lead levels in Airborne by a Portable X-Ray Fluorescence Instrument

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This study was performed to compare the lead levels of 20 quality control standard samples(KOSHA:18-2000) and 72 field samples in lead-acid battery manufacturing plant between ICP and portable-XRF methods.

1. While the proficiencies of 20 quality control standard samples by ICP were 100%, those of analytic result values by XRF were 75%.

2. The correlation coefficient(r) between the reference values for quality control (REF) and the analytic result values by ICP (ICP) was 1.0(p<0.05), and simple linear regression equation and the coefficient(R2) were REF = -0.0009 + 1.016 ICP and 0.9997, respectively.

3. The correlation coefficient(r) between the analytic result values of quality control standard samples by ICP (ICP) and by XRF (XRF) was 0.975(p<0.05), and simple linear regression equation and the coefficient(R2) were ICP = -0.0003 + 1.002 XRF and 0.950, respectively.

4. The correlation coefficient(r) between the analytic result values for lead samples of a lead-acid battery manufacturing plant by ICP (ICP) and by XRF (XRF) was 0.993(p<0.05), and

simple linear regression equation and the coefficient(R2) were ICP = -2.058 + 0.996 XRF and 0.987, respectively.

5. While the frequency distributions of XRF /ICP(Ratio) for each ICP concentration levels in a lead-acid battery manufacturing plant revealed high proportion in ratio range of 0.876-1.125 than in ration range of 1.126-1.375. Also, ICP concentration level in ration range of 0.786-1.125 was increased with increase of frequency distribution of XRF/ ICP.

6. The limit of detection of XRF on lead was determined to be 6.11µg/filter

The data presented in this study indicated that relationship for lead level of quality control samples and field samples in a lead -acid battery manufacturing plant by ICP and portable-XRF methods was proved. The practicing industrial hygienist can use portable-XRF to produce a rapid on-site determination of lead exposure that can immediately be communicated to workers and help identify appropriate levels of personal protection.

Key Words : Lead levels, ICP, XRF

I.

Inductively Coupled Plasma(ICP) Atomic Absorption Spectrometer(AAS)

X-

X-Ray Fluorescence (XRF)

가

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XRF, 10.5, 12.6, 14.8keV detector가
 Cadmium109(¹⁰⁹Cd) Cobalt57 L XRF가
 (⁵⁷Co) shutter가 X-ray gamma-ray
 ray ¹⁰⁹Cd ⁵⁷Co L XRF signal /noise 가
¹⁰⁹Cd ⁵⁷Co XRF noise가
 detector 가
¹⁰⁹Cd ⁵⁷Co K (shell) 3가
 109Cd 가 X-ray fluorescence power
 88.037 kiloelectron volt(keV) gamma ray, ⁵⁷Co 2 gamma-ray
 ray 122.06135keV, 136.47434keV, XRF
 K 가, X-ray ug of lead/cm²
 가 가, X-ray 가 (Morley, 1999).
 (L) 가 XRF
 2 가 X-ray
¹⁰⁹Cd 가 X-ray 22.1 25.0keV
 가 ⁵⁷Co 가 XRF
 14.41302keV XRF ICP
 가, XRF
 X-ray fluorescence()
) K L
 K 88.005keV
 가, ¹⁰⁹Cd ⁵⁷Co 2000
 gamma-ray K 20 72
 K
 74.2keV 85.45 1.
 keV X-ray L
 10.5, 12.6, 14.8keV X-ray

(Gillian Air sampler, USA) 37mm,
 0.8µm cellulose ester membrane filter
 3-piece cassette 2(±0.1)
 L/min 6
 (Gillian calibrator)

2.

1) XRF분석법

가
¹⁰⁹Cd
 X-ray Fluorescence (XL-309 Lead analyser, NITON Corp, Bedford, Massachusetts)
 XRF
 (National Institute for Occupational Safety and Health, NIOSH)
 (Manual of Analytical Methods) No.7702 (NIOSH No 7702, 1998), 6
 Cardboard acetate sleeve(NITON, Bedford, Massachusetts) filter test platform (NITON, Bedford, Massachusetts)
 Figure 1
 M(Middle), T(Top), B(Bottom)
 86

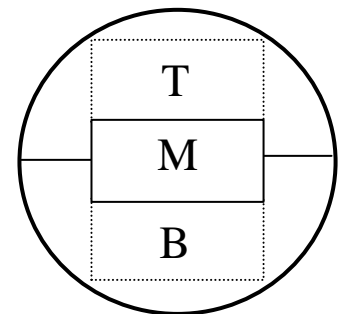


Figure 1. Three readings were taken using a 2cm×1cm aperture

) ug of lead/filter
 = 2.0 × M + 3.8 × T + 3.8 × B

37-mm
 10.75cm²
 9.6cm²
 M
 7.6
 9.6cm²
 2cm²(가) × = 2cm×1cm
 3.8cm²
 T B 3.8cm²

ug of lead/cm² M 18
 2cm² × M (ug of lead/cm²)
 T 3.8cm² × T (ug of
 lead/cm²) B 3.8cm² × B (ug of
 lead/cm²)가 M, T, B
 (Morley, 1996).

2) ICP분석법
 XRF

Glass tube 10ml (No.
 7100, PYREX) 10%
 (MERCK KGaA, Germany) 50-
 60 4-5
 가 XRF
 99%
 No.7300 (NIOSH,
 1994), ICP(ICPS-7500,
 Shimadzu, Japan)
 3.
 SPSS 10.0

ICP XRF

paired t-test

ICP XRF
 (Corre-
 lation analysis)
 ICP
 XRF/ICP(Ratio)
 (Cross tab analy-
 sis)

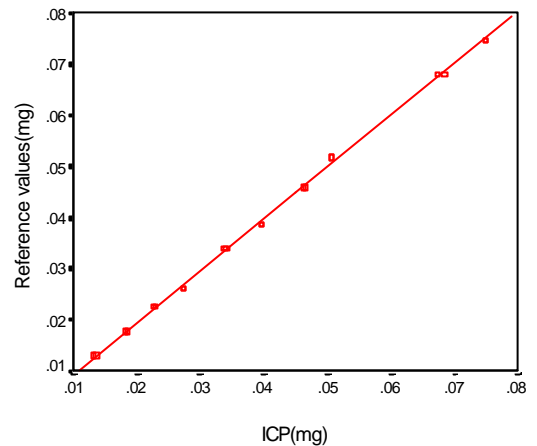


Figure 2. Relationship of reference values and ICP values for lead of quality control samples

2000
 20
 ICP XRF
 , ICP XRF
 Table 1
 ICP 20
 100%
 XRF 15
 ICP
 가

ICP
 R-square 0.9997
 (Correlation) 1.000
 (p < 0.05).

R² = 0.9997
 Correlation = 1.000(p < 0.05)
 Reference values = -0.0009 + 1.016 × ICP

Figure 2

Table 1. Results of analysis for lead of quality control samples by ICP and XRF (unit : mg)

No	ICP	XRF	Reference values	Acceptable range	Results	
					ICP	XRF
1A	0.0748	0.0634	0.0749	0.0659 - 0.0839	P*	N.P [†]
1B	0.0749	0.0688			P	P
2A	0.0132	0.0092	0.0130	0.0103 - 0.0157	P	N.P
2B	0.0137	0.0074			P	N.P
3A	0.0462	0.0455	0.0459	0.0404 - 0.0514	P	P
3B	0.0465	0.0517			P	N.P
4A	0.0228	0.0256	0.0225	0.0189 - 0.0261	P	P
4B	0.0229	0.0247			P	P
5A	0.0342	0.0371	0.0339	0.0291 - 0.0387	P	P
5B	0.0337	0.0379			P	P
6A	0.0397	0.0402	0.0386	0.0335 - 0.0437	P	P
6B	0.0397	0.0380			P	P
7A	0.0273	0.0274	0.0261	0.0213 - 0.0309	P	P
7B	0.0274	0.0332			P	N.P
8A	0.0684	0.0731	0.0680	0.0598 - 0.0762	P	P
8B	0.0672	0.0667			P	P
9A	0.0184	0.0193	0.0178	0.0124 - 0.0232	P	P
9B	0.0182	0.0203			P	P
10A	0.0508	0.0502	0.0519	0.0457 - 0.0581	P	P
10B	0.0508	0.0561			P	P

* P : Proficient

† NP : Not Proficient

	N	Mean(mg)	SD
ICP	20	0.0395	0.0200
Reference values	20	0.0393	0.0203

Figure 3

R-square 0.950
(Correlation) 0.975
(p < 0.05).

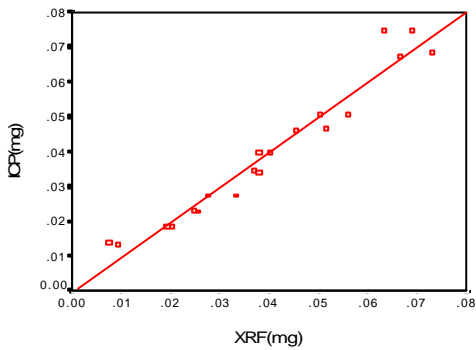


Figure 3. Relationship of XRF values and ICP values for lead of quality control samples

$R^2 = 0.950$
Correlation = 0.975(p < 0.05)
ICP = -0.0003 + 1.002 × XRF

	N	Mean(mg)	SD
ICP	20	0.0395	0.0200
XRF	20	0.0503	0.0247

Figure 4

R-square 0.987
(Correlation) 0.993
(p < 0.05).

$R^2 = 0.987$
Correlation = 0.993(p < 0.05)
ICP = -2.058 + 0.966 × XRF

	N	Mean	SD
ICP	72	91.23	89.51
XRF	72	96.62	87.03

Table 3

ICP XRF

가 0.025
12
5
3
3
1
(Ratio) 1
(8.3%)

ICP
가 0.025-0.049
12

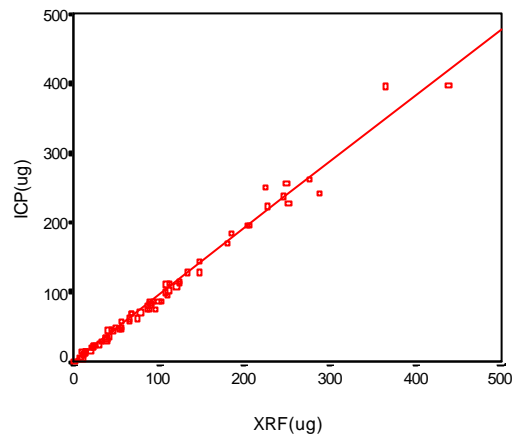


Figure 4. Relationship of XRF values and ICP values for lead of lead-acid battery manufacturing plant

Table 2 Results of analysis for airborne lead in a lead-acid battery manufacturing plant by ICP and XRF

(unit : μg)					
No	XRF	ICP	No	XRF	ICP
1	23.9	19.8	37	133.3	127.7
2	13.2	24.5	38	49.5	48.4
3	24.5	22.0	39	32.9	31.0
4	30.4	26.0	40	77.8	71.6
5	37.2	34.8	41	111.2	102.6
6	42.2	36.6	42	85.8	80.9
7	13.9	14.5	43	86.2	76.2
8	20.9	20.3	44	38.1	31.5
9	8.1	7.1	45	1.6	2.4
10	11.7	12.6	46	74.3	61.8
11	66.5	63.3	47	38.0	30.6
12	123.2	115.5	48	26.2	23.8
13	67.2	68.9	49	68.2	69.4
14	226.9	223.3	50	124.7	113.4
15	179.9	170.0	51	12.4	10.7
16	88.3	86.3	52	10.8	15.4
17	251.5	227.8	53	46.6	43.5
18	57.0	46.8	54	52.4	46.9
19	95.3	76.3	55	11.1	5.8
20	109.1	111.3	56	19.7	15.1
21	113.1	112.7	57	223.6	249.6
22	107.1	99.5	58	103.6	85.9
23	205.8	195.5	59	88.6	76.5
24	14.4	13.8	60	246.0	238.9
25	275.2	262.0	61	439.3	396.8
26	249.6	256.4	62	99.8	85.6
27	23.5	23.2	63	90.3	84.2
28	41.7	45.7	64	93.3	87.0
29	366.5	396.0	65	43.8	47.0
30	90.9	81.1	66	13.3	15.3
31	57.4	48.5	67	15.2	15.6
32	65.2	57.5	68	57.4	57.5
33	120.6	108.8	69	38.8	33.1
34	204.8	195.6	70	147.7	128.8
35	146.7	145.8	71	287.6	241.3
36	184.5	184.2	72	110.4	96.6

0.876-1.125 6 (50.0%), 1.126-1.375 6 (50.0%) . ICP 가 0.050-0.099 16 0.876-1.125 11 (68.8%), 1.126-1.375 5 (31.2%) . ICP 가 0.100 32 0.876-1.125 25 (78.1%), 1.126 -1.375 7 (21.9%) .

LOD(Limit of Detection) 6 가 NIOSH Standard Operating Procedure(SOP)018 3 LOD (NIOSH SOP 018, 1991). 5.11µg , Morely (1999) XRF (700XRF, NITON Corp, Bedford., Massachusetts) 가 NIOSH SOP 018 LOD 6.2 µg (Spike) 가 XRF 90° 가 Morley (1999) XRF 가 65 , ICP XRF ICP 20 100% , XRF 15 75% 가 2. ICP R-square 0.9997 (Correlation) 1.000 (p < 0.05). 3. ICP XRF R-square 0.950 (Correlation) 0.975 (p < 0.05). 4. ICP XRF R-square 0.987 (Correlation) 0.993

Table 3. Frequency distribution of XRF/ICP(Ratio) for each of ICP concentration levels in a lead-acid battery manufacturing plant

Range* (mg/m ³)	XRF/ICP(Ratio)					Total
	0.626-0.875	0.876-1.125	1.126-1.375	1.376-1.625	1.625 <	
< 0.025(%)	3(25.0)	5(41.7)	3(25.0)	0(0.0)	1(8.3)	12
0.025 - 0.049(%)	0(0.0)	6(50.0)	6(50.0)	0(0.0)	1(0.0)	12
0.050 - 0.099(%)	0(0.0)	11(68.8)	5(31.2)	0(0.0)	0(0.0)	16
0.100 (%)	0(0.0)	25(78.1)	7(21.9)	0(0.0)	0(0.0)	32

* Range : Range of ICP concentration levels

0.05).
 5. XRF/ICP(Ratio) ICP
 XRF/ICP(Ratio) 0.876 -
 1.125가
 1.126 - 1.375 .가
 0.876 - 1.125 ICP
 가
 6. XRF LOD
 6.11µg/filter
 XRF
 1
 XRF ICP
 XRF
 가
 가

(p <

XRF

314

REFERENCES

Kiefer M, Morley JC. Cass lake indian Health Service Hospital Cass lake, Minnesota. Hazard Evaluations and Technical Assistance Report No96-209; 1996

Malkin R, Decker J. Philadelphia School district Franklin learning center Philadelphia, Pennsylvania. Hazard Evaluations and Technical Assistance Report No96-0140-2606 ;1996

Morley JC, Clark CS, Deddens JA, Ashely K, Roda S. Evaluation of portable x-ray fluorescence instrument for the determination of lead in workplace air samples. Applied Occupational and Environmental Hygiene 1999;14:306-

National Institute for Occupational Safety and Health(NIOSH). Lead by field portable XRF. NIOSH Manual of Analytical Methods, Method No.7702. NIOSH, Cincinnati, OH:1998

National Institute for Occupational Safety and Health(NIOSH). Elements by ICP, NIOSH Manual of Analytical Methods, Method No.730, NIOSH, Cincinnati, OH:1994

NIOSH. SOP 018 Limits of detection and quantitation. Quality Assurance & Laboratory operation procedures of the Measurements Research Support Branch, Methods Research Branch. Cincinnati, OH: U.S Departments of Health and Human Service, Public Health Service, center for Disease Control, National Institute for Occupational Safety and Health, Division of Physical Science and Engineering. Revision 6, December 1991