

CANMET

REPORT 81-7E

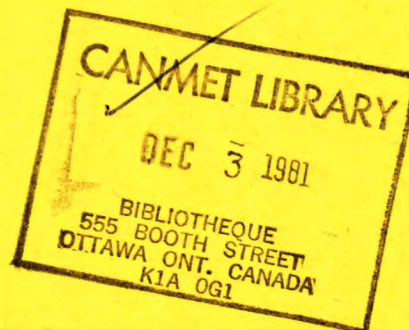
Canada Centre
for Mineral
and Energy
Technology

Centre canadien
de la technologie
des minéraux
et de l'énergie

PD-1: A CERTIFIED NON-FERROUS REFERENCE DUST

H.F. STEGER and W.S. BOWMAN

B.D. McDONNELL AND C. PUPP
ENVIRONMENT CANADA



MINERALS RESEARCH PROGRAM
MINERAL SCIENCES LABORATORIES



Canada



Energy, Mines and
Resources Canada

Énergie, Mines et
Ressources Canada

JULY 1981

© Minister of Supply and Services Canada 1981

© Ministre des Approvisionnements et Services Canada 1981

Available in Canada through

En vente au Canada par l'entremise de nos

Authorized Bookstore Agents
and other bookstores

agents libraires agréés
et autres librairies

or by mail from

ou par la poste au:

Canadian Government Publishing Centre
Supply and Services Canada
Hull, Quebec, Canada K1A 0S9

Centre d'édition du gouvernement du Canada
Approvisionnement et Services Canada
Hull, Québec, Canada K1A 0S9

CANMET

CANMET

Energy, Mines and Resources Canada,
555 Booth St.,
Ottawa, Canada K1A 0G1

Énergie, Mines et Ressources Canada,
555, rue Booth
Ottawa, Canada K1A 0G1

or through your bookseller

ou chez votre libraire.

Catalogue No. M38-13/81-7E

Canada: \$2.00

N° de catalogue M38-13/81-7E

Canada: \$2.00

ISBN 0-660-11046-6

Other countries: \$2.40

ISBN 0-660-11046-6

Hors Canada: \$2.40

Price subject to change without notice.

Prix sujet à changement sans avis préalable.

PD-1: A CERTIFIED NON-FERROUS REFERENCE DUST

by

H.F. Steger*, W.A. Bowman**, B.D. McDonnell⁺ and C. Pupp⁺⁺

SYNOPSIS

A 165-kg sample of non-ferrous dust, PD-1, from the zinc-copper smelter of Hudson Bay Mining and Smelting Company Limited, Flin Flon, Manitoba, was prepared as a compositional reference material in cooperation with the Air Pollution Technology Centre, Environment Canada. PD-1 was ground to minus 74 μm , blended in one lot and tested for homogeneity by chemical methods both before and after bottling in 200-g units.

In a "free-choice" analytical program, 26 laboratories contributed results for one or more of lead, arsenic and mercury in one bottle of PD-1. Based on a statistical analysis of the data, the following recommended values were assigned: Pb, 2.75%; As, 0.77%; and Hg, 389 $\mu\text{g/g}$.

*Research scientist and **Technologist, Mineral Sciences Laboratories, CANMET, Energy, Mines and Resources Canada, Ottawa.

⁺Chemist and ⁺⁺Research Scientist, Air Pollution Technology Centre, Environment Canada, Ottawa.

Note: Major contributions to the certification of PD-1 were also made by other staff members of CANMET and the Air Pollution Technology Centre.

PD-1: Poussière non-ferreuse De Référence Certifiée

par

H.F. Steger*, W.S. Bowman**, B.D. McDonnell⁺ et C. Pupp⁺⁺

SYNOPSIS

Un échantillon de 165 kg de poussière non ferreuse, PD-1, de l'usine de zinc et cuivre de Hudson Bay Mining and Smelting Company Limited, Flin Flon, Manitoba, a été préparé comme matériau de référence de composition en coopération avec le Centre technique de la pollution de l'air, Environnement Canada. Le PD-1 a été broyé à une granulométrie de moins 74 μm , mélangée en lot de la poussière et soumis à des essais d'homogénéité par des méthodes chimiques aussi bien qu'avant et après avoir été embouteillé en unités de 200 g.

En vertu d'un programme analytique de "libre choix", 26 laboratoires ont soumis les résultats d'un flacon de PD-1 pour un ou plus d'un du plomb, arsenic et mercure. Suite à l'analyse statistique des données, les valeurs recommandées suivantes ont été assignées: Pb: 2,75%; As; 0,77% et Hg; 389 $\mu\text{g/g}$.

*Chercheur scientifique et **technologue, Laboratoires des sciences minérales, CANMET, Energie, Mines et Ressources Canada, Ottawa.

⁺Chimiste et ⁺⁺chercheur scientifique, Centre technique de la pollution de l'air, Environnement Canada, Ottawa.

Note: Avec la collaboration d'autres membres du personnel de CANMET et du Centre technique de la pollution de l'air.

CONTENTS

	<u>Page</u>
SYNOPSIS	1
SYNOPSIS	ii
INTRODUCTION	1
NATURE AND PREPARATION	1
INTERLABORATORY PROGRAM FOR CERTIFICATION	2
STATISTICAL TREATMENT OF ANALYTICAL RESULTS	2
Detection of Outliers	2
Estimation of Consensus Values and 95% Confidence Limits	2
DISCUSSION OF ANALYTICAL RESULTS	7
Recommended value for lead	7
REFERENCES	9
APPENDIX A - CONFIRMATION OF HOMOGENEITY	A-11
APPENDIX B - PARTICIPATING LABORATORIES	B-15

TABLES

1. Approximate chemical composition of PD-1	1
2. Particle size analysis	2
3. Recommended values and associated statistical parameters .	2
4a. Summary of analytical results for lead	3
4b. Summary of analytical results for arsenic	4
4c. Summary of analytical results for mercury	5
5a. Analytical results, laboratory means and standard deviation for lead	5
5b. Analytical results, laboratory means and standard deviation for arsenic	6
6. Values of overall mean and median for lead	8
A-1 Confirmation of homogeneity of PD-1	A-13

FIGURES

1. Histogram for lead	8
-----------------------------	---

INTRODUCTION

This report describes the preparation, characterization and certification of non-ferrous dust, PD-1. This work is another example of the continuing endeavor of the Canadian Certified Reference Materials Project (CCRMP) to respond to the need for compositional reference ores, concentrates and related materials typical of Canadian mineral deposits and generally not available elsewhere by analytical laboratories associated with mining, metallurgy, earth sciences and the environment. Other certified reference materials are described in a catalogue available from CANMET, Energy, Mines and Resources Canada, Ottawa (1).

PD-1 is the result of a cooperative effort between CCRMP and the Air Pollution Technology Centre (APTC) of Environment Canada. APTC had initially requested CCRMP to prepare a homogeneous non-ferrous dust from a base metal smelter for use in a nationwide analytical quality assurance program operated by the Federal Provincial Committee on Air Pollution for the benefit of Canadian laboratories concerned with the analysis of similar environmental samples (2). CCRMP suggested, first, that the quantity prepared be increased to provide sufficient reference material for future needs and, second, that PD-1 be certified for lead, arsenic and mercury using the results of laboratories participating in the quality assurance program but which performed the analysis according to the guidelines suggested for the interlaboratory programs of CCRMP.

The interlaboratory program thus led to results for lead, arsenic and mercury from commercial, industrial and government laboratories using analytical methods of their own choice.

NATURE AND PREPARATION

The raw material for PD-1 was donated in April 1980 by Hudson Bay Mining and Smelting Company Limited in Flin Flon, Manitoba. PD-1 is a composite of dusts from numbers 1 and 2 Baghouses and smaller quantities of collected electrostatic

precipitator dusts from the zinc and copper roaster stacks. PD-1 contains varying minor amounts of chalcocite, chalcopyrite, covellite, ferrites, galena, iron oxides, quartz, pyrite, pyrrhotite, sphalerite, elemental sulphur and complex sulphates, silicates and arsenates in a zincite matrix (3).

PD-1 was dry-ground on receipt to pass a 74 μm screen. The dust, weighing 165 kg, was tumbled in a 570-L conical blender for 10 h and eleven samples were taken from pre-selected sites for analysis. These were analyzed in duplicate for copper, lead, zinc, arsenic and mercury by chemical or instrumental methods by APTC. PD-1 was found to be sufficiently homogeneous to qualify as a reference material and was bottled in 200-g units and heat-sealed in polyester-aluminum foil - polyethylene laminated pouches to prevent oxidation of the sulphide and metallic components during storage at CANMET.

The homogeneity of the stock was confirmed for lead. The results are reported in Appendix A.

The approximate chemical composition and particle size analysis are given in Tables 1 and 2.

Table 1 - Approximate chemical composition of PD-1

Element	wt %
Zn	35.9*
Fe	12.20
S (Total)	8.23
S (SO_4^{-2})	4.27
Cu	7.03*
Si	3.05
Pb	2.75 ⁺
As	0.76 ⁺
Cd	0.28
Hg	389 $\mu\text{g/g}^+$
H_2O (105°C)	0.4

*Mean of 22 determinations by APTC. Other values are the mean of duplicate determinations by the Chemical Laboratory, CANMET.

⁺Certified value.

Table 2 - Particle size analysis (wet screen)

Size of fraction (μm)	wt %*
- 104 + 74	0.0
- 74 + 55	0.2
- 55 + 46	12.1
- 46 + 37	8.8
- 37	78.9

*Mean of duplicate determinations.

INTERLABORATORY PROGRAM FOR CERTIFICATION

The laboratories that participated in the certification program are listed in Appendix B. Each was assigned a code number which bore no relation to its alphabetical order.

Each laboratory was requested to contribute five replicate results for lead, arsenic and mercury on one bottle of PD-1 by methods of their choice. Some laboratories however deviated from the request for five results for each element or did not contribute results for all elements. Some laboratories submitted results by more than one method; each set was considered statistically independent.

The recommended values for lead, arsenic and mercury are presented in Table 3. Methodological and statistical information is reported in Tables 4 and 5.

STATISTICAL TREATMENT OF ANALYTICAL RESULTS

DETECTION OF OUTLIERS

Any result obviously suspect for methodological reasons was rejected. Sets of results with unusually high variance were examined and any individual outlying result was deleted. In extreme cases, entire sets with high variance were rejected. Sets of results whose means differed by more than twice the overall standard deviation from the initial mean value were not used in subsequent computations to avoid biasing of the statistics.

ESTIMATION OF CONSENSUS VALUES AND 95%

CONFIDENCE LIMITS

A one-way analysis of variance technique was used to estimate the consensus values and their variance. This approach considers the results of the described certification program to be only one sampling out of a universal set of results. The analytical data were assumed to fit the model (4).

$$x_{ij} = \mu + y_i + e_{ij}$$

where x_{ij} = the j^{th} result in set i ,
 μ = the true consensus value,
 y_i = the discrepancy between the mean of the results in set i (\bar{x}_i) and μ , and

Table 3 - Recommended values and associated statistical parameters (outliers excluded)

Element	Number of laboratories	No. of results	Overall mean	95% CL		σ_A^*
				low	high	
wt %						
Pb	9	45	2.75 ⁺	2.74	2.77	0.01
As	27	131	0.77	0.74	0.79	0.02
Hg	18	88	389 $\mu\text{g/g}$	371	406	14

⁺Select mean - see discussion

*Average within-set standard deviation

e_{ij} = the discrepancy between x_{ij}
and \bar{x}_i

It is assumed that both y_i and e_{ij} are normally distributed with means of zero and variances of ω^2 and σ^2 , respectively. The significance of ω^2 is detected by comparing the ratio of between-set mean squares to within-set mean squares with the F statistic at the 95% confidence level and with the appropriate degrees of freedom.

The consensus value of the assumed model is estimated by the overall mean $\bar{x}_{..}$ by

$$\bar{x}_{..} = \frac{\sum_i \sum_j x_{ij}}{\sum_i n_i}$$

where n_i = the number of results in set i , and
 k = the number of sets.

The value of σ^2 is estimated by s_1^2 which is given by

$$s_1^2 = \frac{\sum_i \sum_j (x_{ij} - \bar{x}_i)^2}{\sum_i n_i - k}$$

The value of ω^2 is estimated by

$$\omega^2 = (s_2^2 - s_1^2) / \frac{1}{k-1} \left(\sum_i n_i - \sum_i n_i^2 / \sum_i n_i \right)$$

where

$$s_2^2 = \frac{\sum_i n_i (\bar{x}_i - \bar{x}_{..})^2}{k-1}$$

The variance of the overall mean is given by

$$V[\bar{x}_{..}] = \left(\sum_i n_i^2 / (\sum_i n_i)^2 \right) \omega^2 + \left(1 / \sum_i n_i \right) \sigma^2$$

and the 95% confidence limits for $x_{..}$ are

$$\bar{x}_{..} \pm t_{0.975, (k-1)} \sqrt{V[\bar{x}_{..}]}$$

It should be noted that the 95% confidence limits denote that if the certification program were performed 100 times, the overall mean in 95 would fall within the prescribed limits.

Table 4a - Summary of analytical results for lead (outliers excluded)

Method	Decomposition, separation, etc.	k	Laboratory number	\bar{x} (wt %)
Atomic absorption	HNO ₃ + one or more of HCl, HF, HClO ₄ , HBr	16	1,4,7,11,16,17,18, 19,20,22,24a,24b, 25,27,28a,29b	2.680
	HNO ₃ + H ₂ SO ₄ + Br ₂ + KBr	1	3	2.748
	HNO ₃	3	12,23,31	2.630
	HCl	1	5	2.420
	Fusion with Na ₂ O ₂ , dissolved in dilute HCl	1	21	2.740
ICP-AES	HNO ₃ + one or more of HCl, HClO ₄	5	26,28b,29a,30,32	2.736
Titrimetry	HF, HNO ₃ , HCl, H ₂ SO ₄ ; PbSO ₄ dissolved in ammonium acetate; titrated with EDTA solution using Hg-drop and calomel electrodes.	1	8	2.733

Table 4b - Summary of analytical results for arsenic (outliers excluded)

Method	Decomposition, separation, etc.	k	Laboratory number	\bar{x} (wt %)
Atomic Absorption	HNO_3 + one or more of HCl , HClO_4 , HF	5	4,7,22,24a,24b	0.775
	HNO_3 + H_2SO_4 + Br_2 + KBr	1	3a	0.778
	1:1 HNO_3 saturated with KClO_3	1	8	0.748
	HNO_3 + one or more of HCl , HClO_4 ; arsine generation	3	16,19,23	0.716
	H_2SO_4 ; arsine generation	1	32	0.704
ICP-AES	HNO_3 + one or more of HCl , HClO_4 , H_2SO_4	4	13,26,28, 29	0.768
DCP-AES	HNO_3 + one or more of HCl , HClO_4 ,	3	24c,24d,24e	0.789
Colorimetry	HNO_3 + one or more of HCl , HClO_4 , H_2SO_4 ; complexing with silver diethyldithiocarbamate	3	1,12a,20	0.849
	HNO_3 + H_2SO_4 ; arsenic distilled as AsCl_3 ; determined as arseno-molybdenum blue complex	2	21,27	0.735
	HNO_3 + H_2SO_4 + Br_2 + KBr ; arsenic distilled as AsCl_3 ; determined as arseno-molybdenum blue complex	1	36	0.754
	HNO_3 + HCl + H_2SO_4 + Br_2 ; arsenic separated as hydrous oxide; reduced to As(III) and extracted as xanthate; re-extracted into water and oxidized to As(V) with Br_2 in CCl_4 ; determined as arseno-molybdenum blue complex.	1	10	0.708
Titrimetry	HNO_3 + HCl + H_2SO_4 ; arsenic distilled as AsCl_3 ; neutralized with NaHCO_3 and titrated with standard I_2 solution	1	12b	0.748
	HNO_3 - HCl - H_2SO_4 + Br_2 ; arsenic reduced with hydrazine sulphate and distilled as AsCl_3 ; neutralized with Na_2CO_3 and titrated with standard I_2 solution	1	17	0.764

Table 4c - Summary of analytical results for mercury (outliers excluded)

Method	Decomposition, separation, etc.	k	Laboratory number	\bar{x} (wt %)
Cold vapour- atomic absorption	HNO ₃ + one or more of HCl, HClO ₄ , H ₂ SO ₄ ,	9	3,4,19,21,22,23,24, 26,31	394
	HBr, Br ₂ , KMnO ₄ , K ₂ S ₂ O ₈ , H ₂ O ₂ ; mercury reduced with hydroxylamine and/or SnCl ₂	9	1,7,8,12,16,20,25 27,28	382
As above; no details on reducing agent				

Table 5a - Analytical results, laboratory means and standard deviation for lead (m %)

					Mean	S.D.
LAB- 1 (AA)	2.72	2.76	2.79	2.74	2.753	.030
LAB- 3 (AA)	2.74	2.75	2.75	2.74	2.75	.008
	2.76					
LAB- 4 (AA)	2.90	2.81	2.83	2.96	2.81	.066
LAB- 5 (AA)	2.49	2.35	2.42		2.420	.072
LAB- 7 (AA)	2.77	2.79	2.79	2.78	2.78	.008
LAB- 8 (TITR)	2.74	2.73	2.73	2.73	2.74	.004
LAB-11 (AA)	2.55	2.52	2.48	2.52	2.58	.037
LAB-12 (AA)	2.60	2.60	2.60	2.60	2.50	.045
LAB-13 (AE) ⁺	2.16	2.22	1.81	2.70	2.89	.435
LAB-16 (AA)	2.76	2.76	2.78	2.79	2.73	.023
LAB-17 (AA)	2.74	2.74	2.74	2.74	2.74	.000
LAB-18 (AA)	2.72	2.78	2.73	2.76	2.76	.024
LAB-19 (AA)	2.76	2.78	2.76	2.76	2.77	.009
LAB-20 (AA)	2.52	2.54	2.47		2.510	.036
LAB-21 (AA)	2.72	2.78	2.73	2.75	2.76	.024
LAB-22 (AA)	2.86	2.88	2.88	2.77	2.86	.044
LAB-23 (AA)	2.70	2.72	2.69	2.65	2.66	.029
LAB-24 (AA)*	2.63	2.33	2.64	2.65	2.66	.141
LAB-24 (AA)	2.60	2.67	2.72	2.65	2.66	.043
LAB-25 (AA)	2.60	2.58	2.55	2.65	2.88	.133
LAB-26 (AE)	2.38	2.49	2.46	2.42	2.47	.040
LAB-27 (AA)	2.59	2.60	2.64	2.60	2.61	.019
LAB-28 (AA)	2.66	2.61	2.67	2.68	2.68	.029
LAB-28 (AE)	2.77	2.83	2.89	2.83	3.01	.091
LAB-29 (AE)	2.53	2.52	2.56		2.535	.022
LAB-29 (AA)*	2.64	2.37	2.30	2.37	2.34	.134
LAB-30 (AE)	2.75	2.79	2.87	2.81	2.88	.055
LAB-31 (AA)	2.64	2.63	2.62	2.64	2.62	.009
LAB-32 (AE)	3.10	2.89	3.08	3.04	2.98	.085

*Single datum identified as outlier

⁺Data set identified as outlier

Table 5b - Analytical results, laboratory means and standard deviation for arsenic (wt %)

						Mean	S.D.
LAB- 1 (COLOR)	.86	.86	.90	.81		.858	.037
LAB- 3 (AA)	.78	.78	.79	.77	.768	.778	.012
	.79						
LAB- 3 (COLOR)	.74	.74	.76	.78	.75	.754	.017
LAB- 4 (AA)	.93	.93	.90			.919	.018
LAB- 7 (AA)	.75	.74	.75	.75	.74	.748	.005
LAB- 8 (AA)	.76	.76	.77	.77	.78	.769	.006
LAB-12 (COLOR)	.82	.83	.70	.68	.71	.748	.071
LAB-12 (TITR)	.75	.75	.75	.74	.74	.748	.004
LAB-13 (AE)	.75	.77	.70	.76	.81	.760	.043
LAB-16 (AA)	.68	.66	.67	.69	.68	.674	.008
LAB-18 (COLOR)	.72	.70	.72	.69	.71	.708	.014
LAB-17 (TITR)	.78	.76	.75	.76	.77	.764	.011
LAB-19 (AA)	.76	.77	.77	.76	.77	.765	.005
LAB-20 (COLOR)	.97	.90	.91			.927	.038
LAB-21 (COLOR)	.75	.76	.75	.75	.76	.754	.006
LAB-22 (AA)	.63	.67	.71	.71	.66	.676	.034
LAB-23 (AA)	.69	.70	.67	.76	.72	.708	.034
LAB-24 (AA)	.85	.86	.85	.86	.85	.854	.005
LAB-24 (AE)	.78	.81	.79	.79	.82	.798	.016
LAB-24 (AE)	.79	.78	.79	.78	.78	.784	.005
LAB-24 (AA)	.84	.81	.82	.83	.81	.822	.013
LAB-24 (AE)	.79	.80	.78	.79	.77	.786	.011
LAB-25 (AA)*	1.16	.88	1.09	.96	1.10	1.038	.115
LAB 25 (AE)	.72	.71	.66	.70	.708	.700	.022
LAB-27 (COLOR)	.70	.72	.70	.72	.74	.716	.017
LAB-28 (AE)	.74	.76	.75	.75	.80	.760	.022
LAB-29 (AE)	.85	.87	.85	.85	.82	.848	.017
LAB-31 (AA)*	.62	.58	.44	.74	.44	.564	.128
LAB-32 (AA)	.78	.76	.71	.62	.65	.704	.069

*Data set identified as outlier

Table 5c - Analytical results, laboratory means and standard deviation for mercury ($\mu\text{g/g}$)

						Mean	S.D.
LAB- 1(AA)	342.	346.	345.	338.		343.	4.
LAB- 3(AA)	434.	486.	445.	482.	456.	460.	20.
LAB- 4(AA)	398.	405.	402.	402.	402.	402.	2.
LAB- 7(AA)	390.	385.	390.	390.	395.	390.	4.
LAB- 8(AA)	367.	358.	367.	350.	380.	364.	11.
LAB-12(AA)*	362.	365.	412.	420.	420.	396.	30.
LAB-13(AA)	508.	461.	438.	552.	517.	495.	46.
LAB-16(AA)	380.	372.	378.	377.	385.	378.	5.
LAB-19(AA)	412.	418.	418.	420.	417.	417.	3.
LAB-20(AA)	412.	393.	406.			404.	10.
LAB-21(AA)	378.	378.	392.	400.	377.	385.	10.
LAB-22(AA)	319.	331.	320.	321.	292.	317.	15.
LAB-23(AA)	420.	390.	400.	430.	440.	416.	21.
LAB-24(AA)	411.	400.	419.	410.	410.	410.	7.
LAB-25(AA)	392.	326.	345.	334.	371.	354.	27.
LAB-26(AA)	379.	410.	397.	376.	370.	386.	17.
LAB-27(AA)	412.	400.	408.	410.	405.	407.	5.
LAB-28(AA)*	373.	384.	427.	400.	415.	400.	22.
LAB-29(AA)*	353.	342.	535.			410.	108.
LAB-30(AA)	695.	489.	597.			594.	103.
LAB-31(AA)	350.	350.	396.	330.	320.	349.	29.
LAB-32(AA)*	576.	561.	564.	585.	511.	559.	29.

*Data set identified as outlier.

The average within-set standard deviation, σ_A , is a measure of the average within-bottle precision as determined by the analytical methods used. The implication exists therefore that a laboratory using a method of average or better reproducibility should obtain individual results for a given certified element with a precision that is at least comparable to the reported value of σ_A .

DISCUSSION OF ANALYTICAL RESULTS

Table 4 illustrates that atomic absorption was the most popular technique employed for lead and arsenic. The Inductively Coupled Plasma-

Atomic Emission Spectrometric technique used by six laboratories has the advantage of giving multi-element determinations. An attempt to detect a statistically significant difference between these two techniques was not made because of the low value of k for ICP-AES.

The cold vapour atomic absorption procedure for mercury was used by all laboratories.

Recommended value for lead

The overall mean and median for lead were calculated to be 2.69 and 2.73%, respectively. A difference of this magnitude between these parameters was not encountered in five interlaboratory programs run previously by CCRMP (Table 6). The histogram for lead (Fig. 1) illustrates the skewed

population of lead results, which of course gives rise to the observed difference between the overall mean and median. Therefore, it was concluded that the overall mean was not the most suitable estimate of the lead content and the following procedure was employed to arrive at the recommended value:

The overall mean and standard deviation were calculated for the results from Laboratories 3, 7, 8, 16, 17, 18 and 21, all of which have

demonstrated their accuracy in the analysis for lead in non-ferrous smelter products in previous CCRMP interlaboratory programs. The nine sets of results, of which the individual means lay within 2σ of the overall mean of the results of these seven laboratories, were included in the calculation of a new overall mean and recommended value of 2.75% with 95% confidence interval of $\pm 0.02\%$, for lead in PD-1.

Table 6 - Values of overall mean and median for lead

Reference material	Grand mean	Grand median	Reference
	wt %		
MP-1	1.88	1.88	5,6
KC-1	6.87	6.87	6,7
CZN-1	7.45	7.44	8
CPB-1	64.74	64.74	9
CCU-1	0.106	0.106	10
PD-1	2.69	2.73	--

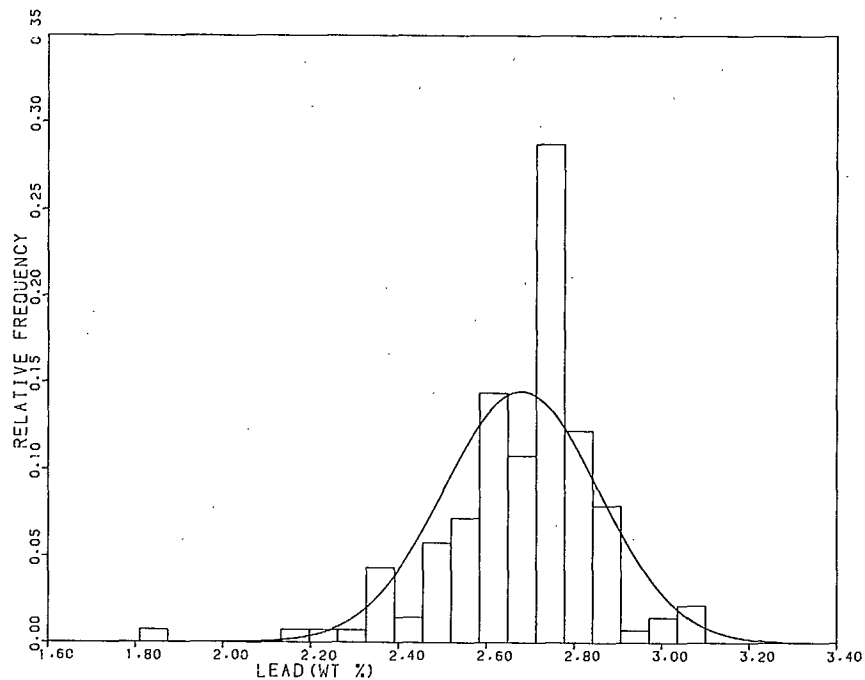
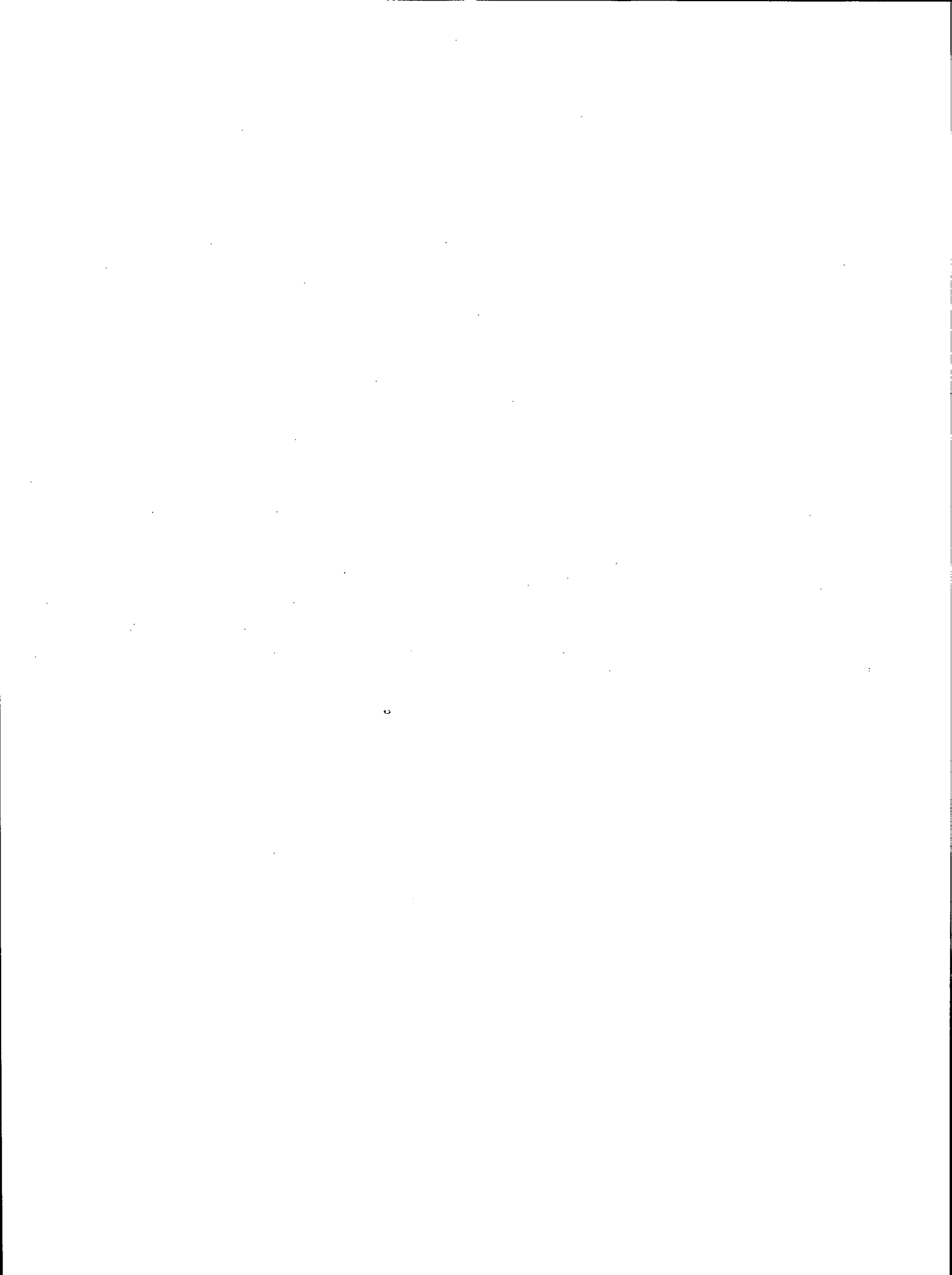


Fig. 1- Histogram for lead

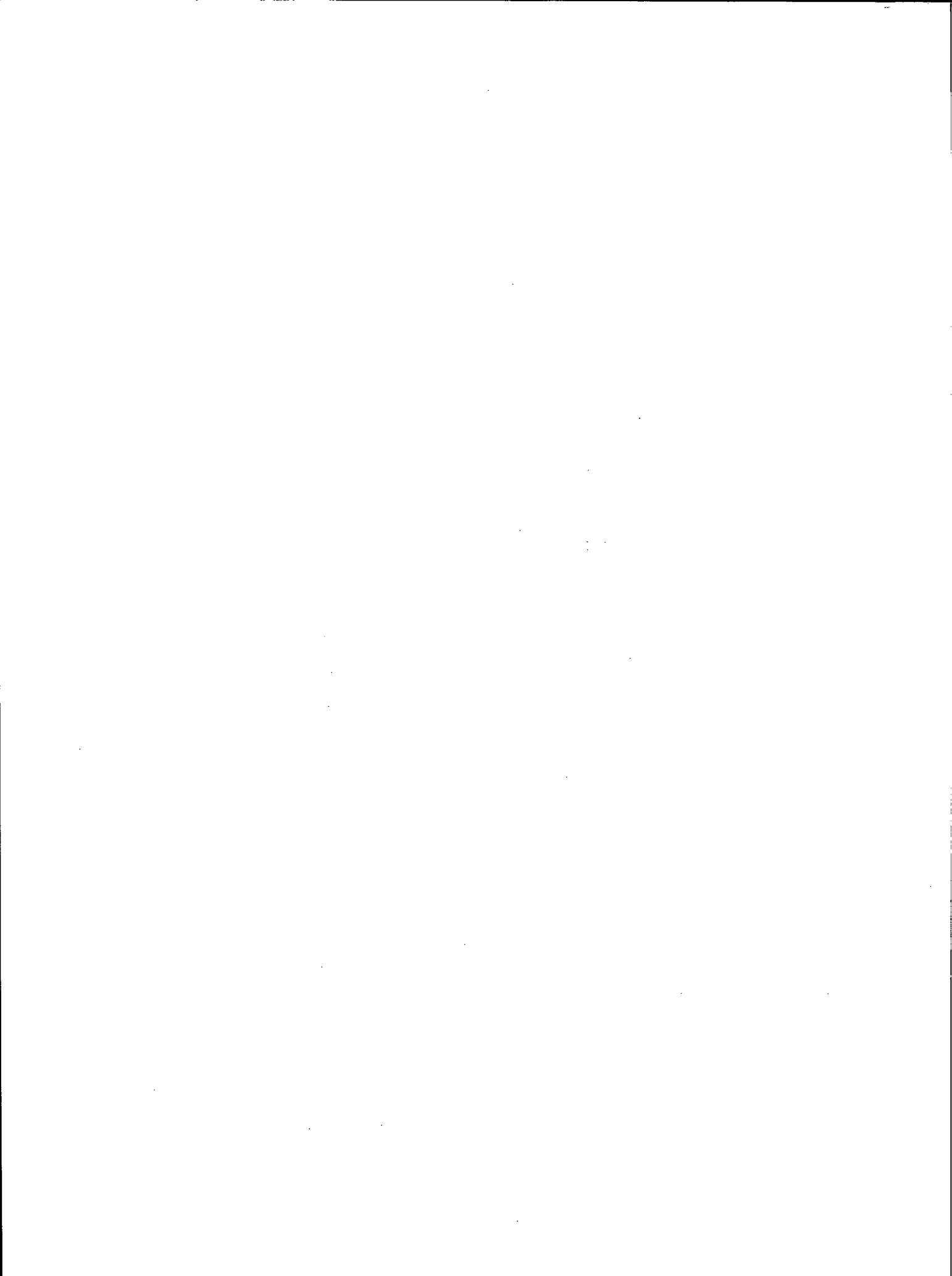
REFERENCES

1. Steger, H.F. "Certified reference materials"; CANMET Report 80-6E; CANMET, Energy, Mines and Resources Canada; 1980.
2. Federal-Provincial Committee on Air Pollution, Record of Meeting, May 27-29, 1980, Appendix F, Environmental Protection Service, Environment Canada.
3. Chen, T.T. (CANMET). Private communication.
4. Brownlee, K.A. "Statistical theory and methodology in science and engineering"; John Wiley and Sons, Inc., New York; 1960.
5. Faye, G.H. "Zinc-tin-copper-lead ore, MP-1: It's characterization and preparation for use as a standard reference material"; Mines Branch Technical Bulletin TB 155; CANMET, Energy, Mines and Resources Canada; 1972.
6. Faye, G.H. and Bowman, W.S. "Revision of Recommended values for reference ores MP-1 and KC-1"; CANMET Report 78-2; CANMET, Energy, Mines and Resources Canada, 1978.
7. Faye, G.H., Bowman, W.S. and Sutarno, R. "Zinc-lead-tin-silver ore KC-1: It's preparation and characterization for use as a certified reference material"; Mines Branch Technical Bulletin TB 193; CANMET, Energy, Mines and Resources Canada, 1974.
8. Faye, G.H., Bowman, W.S. and Sutarno, R. "Zinc concentrate CZN-1 - A certified reference material"; CANMET Report 79-14; CANMET, Energy, Mines and Resources Canada, 1979.
9. Faye, G.H., Bowman, W.A. and Sutarno, R. "Lead concentrate CPB-1 - A certified reference material"; CANMET Report 79-15; CANMET, Energy, Mines and Resources Canada, 1979.
10. Faye, G.H., Bowman, W.S. and Sutarno, R. "Copper concentrate CCU-1 - A certified reference material"; CANMET Report 79-16; CANMET, Energy, Mines and Resources Canada, 1979.



APPENDIX A

CONFIRMATION OF HOMOGENEITY



CONFIRMATION OF HOMOGENEITY

Fifteen bottles of PD-1 were selected out of the stock of 780 for analysis in triplicate for lead by APTC to confirm homogeneity. The stock was first divided into 15 lots of 52 bottles each. The code number of the first bottle was selected at random out of the first lot. The code numbers of the remaining bottles selected were given by the code number of the preceding bottle plus 52. The results are shown in Table 7.

A one-way analysis of variance technique was used to assess the homogeneity (4). Herein, the ratio of the between-bottle to within-bottle mean square is compared with the F statistic at the 95% level of probability. No evidence of bottle to bottle inhomogeneity was found for lead (Table A-1).

Table A-1 - Confirmation of homogeneity of PD-1

Bottle No.	Pb (wt %)			
	Individual			Mean
5	2.76	2.76	2.81	2.78
57	2.79	2.68	2.82	2.76
109	2.73	2.75	2.75	2.74
161	2.79	2.79	2.81	2.80
213	2.84	2.72	2.72	2.76
265	2.71	2.83	2.70	2.75
317	2.82	2.78	2.75	2.78
369	2.80	2.76	2.74	2.77
421	2.77	2.77	2.80	2.78
473	2.76	2.70	2.83	2.76
525	2.80	2.74	2.76	2.77
577	2.76	2.81	2.72	2.76
629	2.76	2.80	2.75	2.77
681	2.76	2.77	2.72	2.75
733	2.79	2.72	2.75	2.75

Overall mean = 2.766

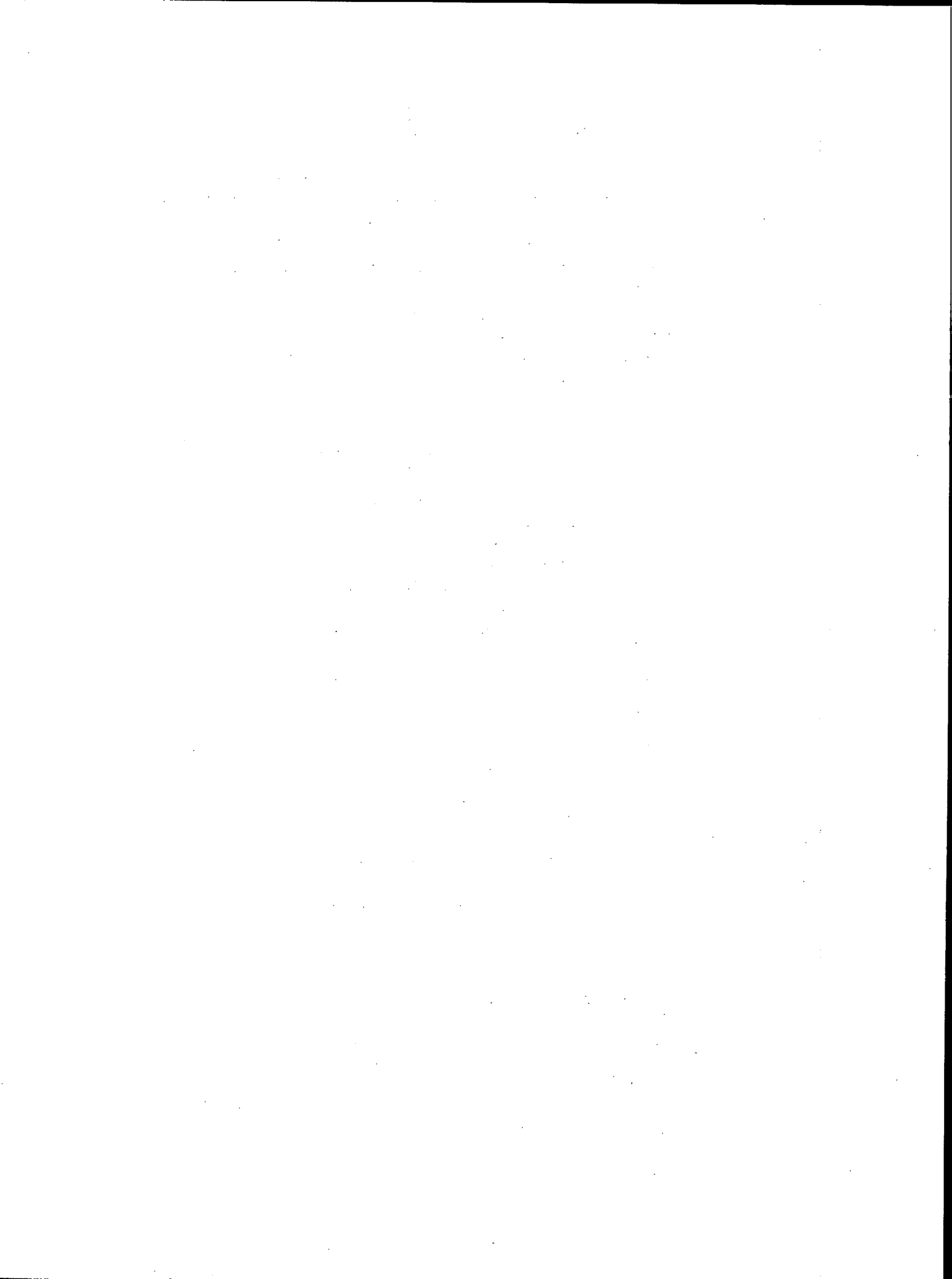
Analysis of Variance Table

<u>Source of variation</u>	<u>Degrees of freedom</u>	<u>Mean square</u>
Between bottles	14	6.27×10^{-4}
Within bottles	30	1.924×10^{-3}
Total	44	

Calculated F statistic = 0.3258

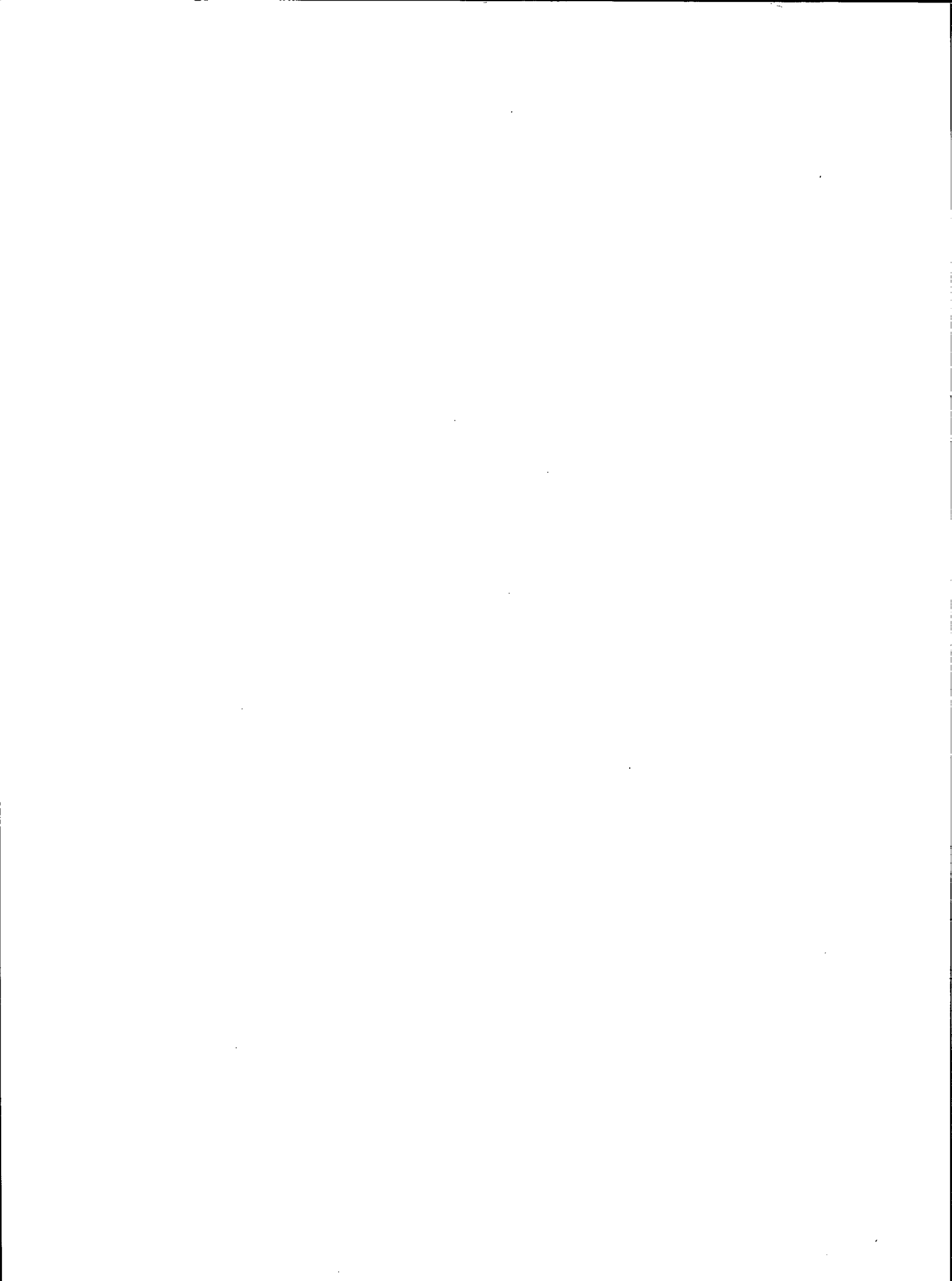
F.95 (14,30) = 2.0374

Null hypothesis of no difference between bottles is accepted.



APPENDIX B

PARTICIPATING LABORATORIES



PARTICIPATING LABORATORIES

Atlantic Analytical Services Ltd.
Springdale, Newfoundland

Barringer Magenta Ltd.
Rexdale, Ontario

Can Test Limited
Vancouver, British Columbia

Centre de Recherche Noranda
Pointe Claire, Quebec

Chemex
Calgary, Alberta

Chemical and Geological Lab. Ltd.
Edmonton, Alberta

CIP Research Ltd.
Hawkesbury, Ontario

Econotech Services Ltd.
New Westminster, British Columbia

Energy, Mines and Resources Canada
CANMET, Ottawa, Ontario

Enviroclean Ltd.
London, Ontario

Environment Canada
EPS, Air Pollution Technology Centre
Ottawa, Ontario

Environment Canada
EPS, Halifax, Nova Scotia

Environment Canada
EPS, West Vancouver
British Columbia

Falconbridge Nickel Mines
Metallurgical Laboratories
Thornhill, Ontario

Falconbridge Nickel Mines
Sudbury Operations
Falconbridge, Ontario

Hudson Bay Mining & Smelting Co. Ltd.
Flin Flon, Manitoba

Inco Metals Ltd.
Geological Research
Copper Cliff, Ontario

Inco Metals Ltd
Manitoba Division
Thompson, Manitoba

Inco Metals Ltd.
J. Roy Gordon Research Laboratory
Sheridan Park, Mississauga,, Ontario

Manitoba Department of Commerce and
Corporate Affairs and Environment
Technical Service Lab
Winnipeg, Manitoba

Ministère de l'Environnement (Québec)
Direction Générale des Inventaires et
de la Recherche
Montréal, Québec

Ontario Research Foundation
Sheridan Park, Mississauga, Ontario

Province of British Columbia
Ministry of Environment
Environmental Laboratory
Vancouver, British Columbia

Province of Ontario
Ministry of the Environment
Laboratory Services Branch
Rexdale, Ontario

Saskatchewan Research Council
Analytical Laboratory
Saskatoon, Saskatchewan

Technical Service Laboratories
Mississauga, Ontario

