National Institute of Occupational Health

Proficiency Testing for Measurement of Total Mass and Elements in Workplace Air Filters. Round 10

Authors: Kari Dahl
Nils Petter Skaugset
Yngvar Thomassen

Co-ordinator: Kari Dahl

Date: 04.06.2003

Series: STAMI Report Vol. 4, No. 3 (2003) **ISSN:**1502-0932

Summary:

In Round 10 of the proficiency testing programme sixteen laboratories from England, Lithuania, Finland, Sweden, Denmark and Norway have participated.

Filter and cassettes were distributed to the laboratories in April 2002. The laboratories were asked to pre-weigh the filters prior to exposure to welding fume, and return the prepared filter cassettes by 10th of May 2002. Realistic workroom air and synthetically produced reference filters were distributed to the participants in June 2002. The deadline for reporting results was 9th of August 2002.

The laboratories were asked to measure a number of occupational important elements listed in the enclosed protocol (Ag, Cd, Cr, Cu, Fe, Mn, Ni, Pb, Zn) and total mass.

The inter-laboratory variance for the individual elements after rejection of outliers shows acceptable agreement among the participants.

Stikkord: Interkalibrering, grunnstoffbestemmelse, gravimetri, arbeidsatmosfære

Key terms: Proficiency testing, elements, total mass, workroom air filters

CONTENTS

SUMMARY

The purpose of this proficiency-testing programme is to assess the performance of methods used for routine measurements by commercial, public and industrial laboratories.

Filters and cassettes were distributed to the laboratories in April 2002. The laboratories were asked to pre-weigh the filters prior to exposure to welding fume, and to return the prepared filter cassettes by 10th of May 2002. Welding fume filters (Series X) and synthetically produced reference filters (Series E) were distributed to the participating laboratories in June 2002 with a deadline of reply 9th of August 2002.

In order to determine the "true" quantities of total mass and elements on the filters, randomly selected parallel filters from each filter series were analysed at the National Institute of Occupational Health in Oslo. The reference values for Series X (welding fume) were based on the results using ICP-OES. The reference values for Series E (reference filters) were calculated and the theoretical values verified by analysis using ICP-OES.

In this round of the proficiency- testing-programme sixteen laboratories from England, Lithuania, Finland, Sweden, Denmark and Norway have participated. Each laboratory was asked to determine a total of nine elements (Ag, Cd, Cr, Cu, Fe, Mn, Ni, Pb, Zn) in two filter matrices and total mass on the welding fume filters.

The inter-laboratory variance for the individual elements after rejection of outliers shows acceptable agreement among the participants.

ABBREVIATIONS

EAAS:	Electrothermal Atomic Absorption Spectrometry
FAAS:	Flame Atomic Absorption Spectrometry
ICP-OES:	Inductively Coupled Plasma Optical Emission Spectrometry
ICP-QMS:	Inductively Coupled Plasma Quadruple Mass Spectrometry
ICP-MS:	Inductively Coupled Plasma Mass Spectrometry
ICP-SFMS:	Inductively Coupled Plasma Sector Field Mass Spectrometry
OEL:	Occupational Exposure Limit
RSD:	Relative Standard Deviation
SD:	Standard Deviation

1. INTRODUCTION

The National Institute of Occupational Health in Oslo is the national reference laboratory for work environment measurements in Norway. As a national reference laboratory one of the objectives is to perform proficiency testing programmes for work environment measurements in commercial, public and industrial laboratories.

There is no official approval scheme for laboratories which offer work environment measurement services in Norway, but the Governmental Labour Inspectorate requests all laboratories to participate in the proficiency testing programmes organised by the National Institute of Occupational Health. Participation is voluntary, and the laboratories are informed in advance that the results will be published with identification of the participants. The purpose of this proficiency-testing programme is to assess the laboratory performance using routine procedures. The laboratories were therefore requested to include the samples in their normal analytical routine.

As in the previous round measurement of total mass was included in the testing programme. Filters and cassettes were distributed to the laboratories in April 2002. The laboratories were asked to pre-weigh the filters prior to exposure to welding fume, and to return the prepared filter cassettes by 10th of May 2002. The prepared samples were distributed in June 2002, with a deadline for replies of 9th of august. Each participant received duplicates of workroom air filters (Series X), reference filters spiked with known quantities of selected elements (Series E) and blank filters. The laboratories were asked to measure total mass (Series X) and the elements listed in the enclosed protocol for both set of filters.

2. PARTICIPATING LABORATORIES

Lab.no	Name, address	Name used
1	Høgskolen i Agder, Analyselaboratoriet Serviceboks 422, 4604 Kristiansand	HiA
2	Analytica AB Aurorum 10, S-977 75 Luleå, Sverige	Analytica AB
3	Centre of Occupational Medicine, Institute of Hygiene Etmonu str. 3, LT-2001 Vilinius, Lithuania	Inst. of Hygiene
4	Falconbridge Nikkelverk A/S, Hovedlaboratoriet P.O.Box 457, N-4601 Kristiansand, Norway	Falconbridge
5	Health & Safety Executive, Inorganic Substances Section Broad Lane, GB-Sheffield S3 7HQ, UK	HSE
6	Hydro Aluminium Karmøy metallverk, 4265 Håvik	HAK
7	Kuopio Region Institut för Arbetshygien P.O.Box 93, SF-70701 Kuopio, Finland	Kuopio
8	Eurofins Danmark A/S Smedeskovvej 38, DK-8464 Galten, Denmark	Eurofins
9	MOLAB as P.O.Box 5000, N-8601 Mo i Rana	Molab
10	Norsk Institutt for Luftforskning Postboks 100, N-2027 Kjeller	NILU
11	Oulun Aluetyöterveyslaitos, Kemian Laboratorio Aapistie 1, SF-90220 Oulu, Finland	Oulu
12	Sero AS Postboks 24, N-1374 Billingstad	Sero
13	Telemark Sentralsjukhus, Yrkesmedisinsk avdeling Ulefossveien, 3710 Skien	TSSH
14	Tinfos as, avd. Øye Smelteverk Postboks 246, 4481 Kvinesdal	Tinfos
15	X-lab Ibsensgt. 104, 5052 Bergen	X-lab
16	Universitetssjukhuset Örebro, Yrkes-och miljömedicinska kliniken, Analyslaboratoriet, S-701 85 Örebro	Örebro

3. WELDING FUME FILTERS

The 114-multi-channel sampler unit used for the collection of replicate filter samples of welding fumes was developed at the National Institute of Occupational Health in Oslo and has been used in previous rounds. The multi-channel sampler unit is described in earlier reports.

3-piece 25-mm plastic filter holders with an extended connecting piece (Omega Speciality Instruments Co. art.no A-002550-3) were used. Particulate matter was collected on 0.8 µm mixed cellulose esters membrane filters (Millipore art.no. AAWP002500).

To ensure constant rate of airflow through the filters during the sampling period each position is equipped with a critical orifice. The rate of flow through each filter was measured at the beginning and at the end of the sampling period using a high precision flow meter in order to calculate an exact sampling air volume for each filter.

In order to obtain homogeneous deposition on the filters the filter holders are open-faced. This is of particular importance for X-ray fluorescence spectrometry for the direct measurement of the analytes.

Welding fumes were generated in the workshop at the National Institute of Occupational Health in Oslo using a variety of welding rods.

4. REFERENCE FILTERS

Reference filters were prepared by spiking 37 mm 0.8 µm mixed cellulose esters membrane filters (Millipore art.no. AAWP003700) with an aqueous solution containing elements with concentrations gravimetrically traceable to ultrapure metals or stoichiometrically well defined oxides. The reference values are based on a gravimetric procedure, i.e. weight per volume composition of the primary reference material dissolved in high purity sub-distilled acids. The spiked analyte masses were measured by weighing each filter. Exact masses of the analytes of individual filters are obtained by using a gravimetric correction factor.

5. ANALYTICAL CONDITIONS

The sample preparation and analytical methods described by the participants are presented in the following table:

No.	Laboratory	Sample Preparation	Sample volume	Analytical method/Instrumentation
1	HiA	The filters were added 4 ml HNO_3/HCl, vaporised to dryness, and added 5 ml HNO_3	50 ml	Varian Spectra AA 220 FS Mettler AE 240
2	Analytica AB	HNO_3/H_2O_2, in Teflon autoclave with microwave assisted digestion.		EPA 200.7 Modified ICP-OES and EPA 200.8 ICP-SFMS
3	Inst. of Hygiene	HNO_3/HCl, Teflon autoclave with microwave assisted digestion		EAAS with Zeeman and chem.mod. Balance SCALTEC SBC 21
4	Falconbridge	2 ml H_2O, 2 ml HCl, 1 ml HNO_3 on hot plate, 10 ml HCl before final dilution to sample volume	50 ml	TJA IRIS ICP-OES Mettler 250
5	HSE	Microwave digestion using HNO_3/HCl/HF		Method based upon BS EN ISO 15202-3, PE Optima 3000 DV ICP-OES
6	HAK			Only gravimetic determination
7	Kuopio	HNO_3/HCl, Teflon autoclave with microwave assisted digestion		PE Analyst 800 Flame Mettler Toledo AT 261
8	Eurofins	Microwave digestion using HNO_3		ICP-OES Micro Balance
9	Molab	Microwave digestion using HNO_3/HCl/HF		ICP-OES NS 4860 (gravimetric determination)
10	NILU	HNO_3/H_2O_2, with microwave assisted digestion.		ICP-MS
11	Oulu	Microwave digestion using HNO_3 and HCl	25 ml	Flame AAS Perkin Elmer 5100 Mettler AT261 DeltaRange
12	Sero	HNO_3/HCl/HF digestion in Teflon autoclaves	14 ml	ICP-OES Perkin Elmer Plasma 2000
13	TSSH			Only gravimetic determination
14	Tinfos	Microwave digestion using HNO_3 and HCl	100 ml	FAAS - Perkin Elmer 3030 Mettler AE 163
15	X-lab	HNO_3/H_2O_2, with microwave assisted digestion, dilution with 2 % NH_4Cl	25 ml	FAAS Metller AT261 DeltaRange
16	Örebro	HNO_3/H_2O_2, with microwave assisted digestion.	25 ml	ICP-QMS Micro Balance Cahn 031

6. REFERENCE VALUES

In order to determine the "true" quantities of elements on the filters, randomly selected parallel filters from each filter series were analysed at the National Institute of Occupational Health in Oslo. Filters from both series were dissolved in 2 ml aqua regia and 0.2 ml hydrofluoric acid in Teflon autoclaves with microwave assisted digestion. After cooling to room temperature all samples were diluted with ultra pure water to a volume of 14 ml.

All volumetric equipment that was used for the preparation of samples and standard solutions was volumetrically calibrated. The maximum volumetric uncertainty was ±0.1 %.

All standard solutions (traceable to NIST primary certified solutions) used for instrument calibrations were matrix-matched to be as nearly as possible identical to the sample solutions in order to minimise inter-element and matrix effects.

A semi-micro-balance of type Sartorius MC 210 P was used for the measurement of total mass. For the simultaneous measurement of all elements a Perkin-Elmer OPTIMA 3000 inductively coupled plasma optical emission spectrometer (ICP-OES) was used.

The reference values for Series X (welding fumes) are based on the results using ICP-OES (elements) and semi-micro-balance (total mass). Correction factors due to differences in sampling air volumes were used.

The spiked analyte masses of the reference filters (Series E) are measured by weighing. Exact values of individual filters are obtained by using a correction factor for each filter. The theoretical values are verified by results using ICP-OES.

The results and reference values for Series X (welding fumes) are given in Appendix 1, table 1 and 2 and the results and reference values for Series E (reference filters) are given in Appendix 1, table 3 and 4.

7. ASSESSMENT CRITERIA

The National Institute of Occupational Health in Oslo has drawn up proposals for assessing analytical performance. Routine measurements of workroom air filters should comply with the following criteria:

Quantity in relation to OEL	Limit 1	Limit 2
≥100 %	Better than ± 5 %	Better than ± 10 %
10 %	Better than ± 10 %	Better than ± 20 %
1 %	Better than ± 25 %	Better than ± 50 %

Accuracy considered «good» or «acceptable» are dependent on the relationship between the concentration in a sample and the occupational exposure limit (OEL) for each individual element, expressed by the following formulas:

Limit 1(in %): $\log y = 4.8 * 10^{-2} * \log x^2 - 4.5 * 10^{-1} * \log x + 1.4$

where x is the proportion of element in sample relative to OEL (in %)

Limit 2 (in %): Limit 1∗2

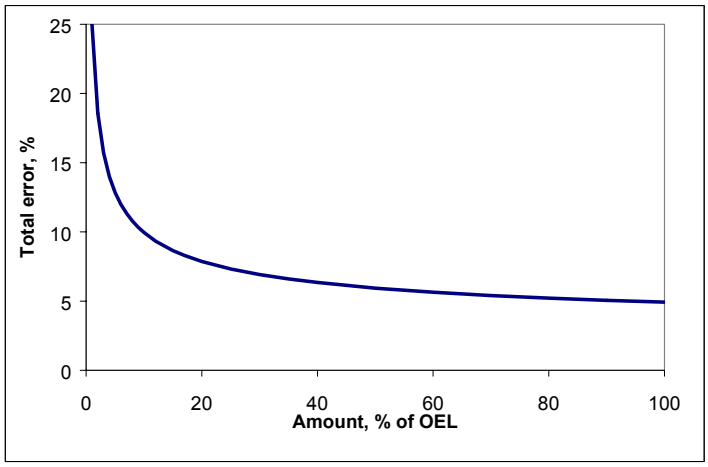


Fig. 1 Plot of the expression for limit 1

In order to take filter homogeneity into account, two times the relative standard deviation of the measurements made by NIOH is added.

The following requirements emerge:

Requirement 1= Limit 1 + filter homogeneity (2RSD)

Requirement 2= Limit 2 + filter homogeneity (2RSD)

Analysis performed at the NIOH shows filter-to-filter varies between 0.2 - 1.0 % (RSD) for Series E and between 1.1 - 4.0 % (RSD) for Series X.
The RSD for each element and the calculations of limits and requirements for Series E and Series X are given in Appendix, table 9 and 10.

8. DETECTION LIMIT

With regard to samples from workroom atmospheres, detection limits of analytical procedures should reflect the threshold limit value for each element. Provided that the filter has been exposed to one cubic meter of air, the detection limit of the applied method of measurements must be no higher than 1% of the OEL.

Element	Norwegian occupational exposure limit, µg/m^3	Recommended detection limit, µg
Ag	100 (metal dust and fume)	1
Cd	20 (cadmium oxide calculated as Cd	0.2
Cr	500 (Cr(II) and Cr(III) calculated as Cr)	5
Cu	100 (fume)	1
Fe	3000 (calculated as Fe)	30
Mn	1000 (fume calculated as Mn)	10
Ni	50 (calculated as Ni)	0.5
Pb	50 (metal dust and fume calculated as Pb)	0.5
Ti	5000 (titanium dioxide) (calculated as Ti 3000 g/m^3)	30
Zn	5000 (zinc oxide) (calculated as Zn 4000 µg/m^3)	4
Total mass	5000 (welding fume)	50

9. RESULTS

The results that are reported by the participating laboratories are given in Appendix 1, Table 3 (Series E) and Table 4 (Series X).

The individual results are also presented graphically in Appendix 2.

The performances of the participating laboratories are summarised in Table 1 and 2.

The following symbols are used:

- Results complying with Requirement 1 («good accuracy») are indicated by: ●
- Results complying with Requirement 2 («acceptable accuracy») are indicated by: ❍
- Results outside these two acceptance limits are indicated «not acceptable»: ↙

To comply with either Requirement 1 or Requirement 2 both parallel measurements must fall within the acceptance limits. Values that coincide with Requirement 1 or Requirement 2 are indicated by ● or ❍

Table 1. Summary of results: Series E - Reference filters.

		Cd	Cr	Cu	Fe	Mn	Ni	Pb	Zn
	Reference value, µg	33.6	170	99.6	341	97.0	37.2	32.0	313
1	HiA	❍	↙	●	●	●	●	❍	●
2	Analytica AB	❍	●	●	●	●	●	❍	●
3	Inst. of Hygiene	↙	❍	↙	↙	●	↙	↙	
4	Falconbridge	●	❍	●	●	●	●	●	●
5	HSE	❍	●	❍	❍	●	❍	❍	●
6	HAK								
7	Kuopio	❍	↙	●	●	●	↙	●	●
8	Eurofins	❍	❍	↙	●	❍	↙	❍	❍
9	Molab	❍	❍	●	●	●	●	●	●
10	NILU	●	●	●	●	●	●	❍	●
11	Oulu	●	●	❍	●	●	●	❍	●
12	Sero	●	●	●	●	●	●	●	●
13	TSSH								
14	Tinfos	●	●	●	●	●	●	●	●
15	X-lab	❍	●	❍	●	●	●	●	●
16	Örebro	●	●	●	●	●	●	●	↙

●: «good accuracy» ❍: «acceptable accuracy» ↙: «not accepted» «blank»: «not measured»

Table 2. Summary of results: Series X - Welding fume filters.

		Total mass	Ag	Cd	Cr	Fe	Mn	Ni	Pb
	Reference value, µg	3150	15.0	2.28	63.3	313	128	10.7	787
1	HiA	●		●	●	●	●	●	↙
2	Analytica AB	●		●	●	●	●	❍	❍
3	Inst. of Hygiene	●		●	●	↙	↙	↙	↙
4	Falconbridge	●	●	●	❍	●	●	❍	●
5	HSE			●	❍	❍	❍	❍	↙
6	HAK	●							
7	Kuopio	●		●	❍	●	●	❍	●
8	Eurofins	●	↙	●	●	●	●	●	●
9	Molab	●		●	●	●	●	●	●
10	NILU	●	↙	●	↙	↙	●	❍	❍
11	Oulu	●		●	●	●	●	●	↙
12	Sero		●	●	●	●	●	●	●
13	TSSH	●							
14	Tinfos	●	●	●	●	●	●	●	●
15	X-lab	●		●	●	●	●	●	●
16	Örebro	●		↙	●	●	●	●	●

●: «good accuracy» ❍: «acceptable accuracy» ↙: «not accepted» «blank»: «not measured»

10. DISCUSSION

In this round of the proficiency testing programme the participating laboratories were asked to determine a total of nine elements in two filter matrices in addition to total mass on welding fume filters. It should be noted that two of the laboratories only wanted to participate in determination of total mass on the welding fume filters. The participating laboratories were asked to mount the filters in the filter cassettes for sampling according to their routine procedure before returning them to NIOH for sampling of welding fume. All filter cassettes that were sent in by the participating laboratories were tested according to NIOH's leak test procedure to prevent air by-pass in the filter cassettes. Filter cassettes which did not comply to the leak test requirements were pressed and re-tested together with the rest of the filter cassettes to be exposed using the multi-sampler-unit. About half of the participating laboratories returned filter cassettes that did not comply to NIOH's leak test requirements.

The parallel sampler was designed for use with 25 mm plastic filter holders (Costar - Nuclepore art. no N-800932) with an extended connecting piece. These filter holders are no longer available and in the two previous rounds the 25 mm plastic filter (Millipore art. no M000025A0) filter holders were mounted to the parallel sampler using external connecting pieces. Problems with leakage during flow measurement using an external connecting piece made it necessary to look for other filter cassettes to avoid these problems. Before this round a provider of 25 mm plastic filter holders with an extended connecting piece (Omega Speciality Instruments Co. art. no A-002550-3) were found and these filter holders were used**.**

Three of the participating laboratories reported blacking of pads in the filter cassettes and control of the rest of the series X showed that about 10 % of the filter cassettes had cracks in the middle section (See fig. 2). These cassettes were tested according to our laboratory procedure and found to be without leakage according to NIOH's leaking test procedure before mounting them to the multi-sampler-unit, but must have cracked before or during the sampling period. Before next round of the proficiency-testing program NIOH will develop a new external-connecting piece for the multi-sampler unit and filter cassettes used in daily routine for air sampling by NIOH will be used.

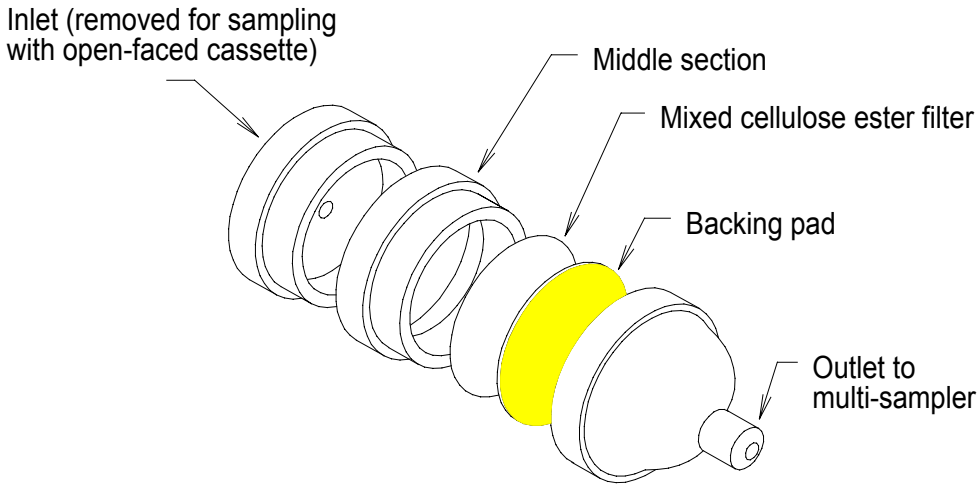


Fig. 2. The 3-piece 25 mm filter holder (Omega Speciality Instruments Co. art. no A-002550-3) used in Round 10.

The inter-laboratory relative standard deviation after rejection of outliers, depending on the element, varies from 2.9 to 11.8 % (3.8 to 18 % in Round 9). In average for all elements the deviation is 6.8 % (8.7 % in Round 9) which shows acceptable agreement among the participants.

Quality control filters for daily use are available from the National Institute of Occupational Health, Oslo, at moderate cost. The use of these may be beneficial in further improving the quality of the laboratory measurements.

Table 3 summarises the results of this proficiency-testing program.

Table 3. Laboratory results for the last ten proficiency-testing programmes.

Round	No of laboratories	No of analytes	No of reported results	● %	❍ %	↙ %	Extreme Values, %
0	9	15	185	65	21	14	12
1	14	22	652	56	24	20	7
2	12	13	372	70	17	10	4
3	18	11	285	68	18	13	2
4	20	11	301	70	14	15	10
5	15	9	199	79	8	13	3
6	16	10	153	78	15	7	2
7	10	10	115	88	6	7	4
8	13	16	152	87	5	8	4
9	12	16	171	73	15	12	2
10	16	16	214	72	17	11	4

APPENDIX 1

Table 1. ICP-OES measurements of welding fume filters, Series X. Randomly selected filters analysed at the National Institute of Occupational Health, Oslo

Analytical Wave-length in nm	Filter no. X017 µg	Filter no. X018 µg	Filter no. X020 µg	Filter no. X022 µg	Filter no. X024 µg	Filter no. X026 µg	Filter no. X027 µg	Filter no. X028 µg	Filter no. X029 µg	Filter no. X030 µg
Ag 328.068	14.8	15.5	15.6	15.4	14.7	14.7	15.0	14.8	14.9	15.2
Ag 338.289	15.0	15.2	14.9	15.3	14.9	14.9	15.1	14.6	15.0	14.9
Cd 214.438	2.15	2.17	2.16	2.28	2.27	2.33	2.35	2.34	2.40	2.36
Cd 226.502	2.14	2.15	2.14	2.28	2.26	2.31	2.34	2.33	2.40	2.35
Cr 205.552	63.9	63.9	62.9	65.0	62.6	62.8	64.3	62.6	64.2	63.4
Cr 206.149	64.2	64.2	63.2	65.2	62.9	63.2	64.7	63.0	64.6	63.8
Cr 357.869	62.7	63.0	61.6	63.7	61.3	61.4	63.2	61.0	63.0	62.2
Fe 234.349	309	311	308	319	302	296	321	314	312	316
Fe 238.204	313	316	312	323	307	299	325	318	317	320
Fe 259.940	313	317	313	322	306	300	325	318	316	321
Fe 239.562	311	314	310	321	305	296	322	316	315	319
Mn 257.610	127	129	128	131	126	127	130	127	128	129
Mn 260.569	127	129	127	131	126	126	130	127	128	129
Mn 294.920	127	129	127	130	125	126	130	127	128	129
Ni 231.604	11.0	11.1	10.8	11.2	10.6	10.7	10.9	10.6	10.9	10.9
Ni 341.476	10.8	11.0	10.5	10.9	10.4	10.5	10.7	10.4	10.7	10.6
Pb 220.353	787	798	791	794	783	769	796	782	779	789

Table 2. Reference values, Series X - welding fume filters.

Filter no.	Total mass µg	Ag µg	Cd µg	Cr µg	Fe µg	Mn µg	Ni µg	Pb µg
X017	3170	14.9	2.15	63.6	311	127	10.9	787
X018	3070	15.3	2.16	63.7	314	129	11.0	798
X020	3120	15.2	2.15	62.6	311	127	10.6	791
X022	3150	15.4	2.28	64.6	321	130	11.0	794
X024	3170	14.8	2.26	62.3	305	126	10.5	783
X026	3210	14.8	2.32	62.5	298	127	10.6	769
X027	3230	15.1	2.34	64.1	323	130	10.8	796
X028	3140	14.7	2.33	62.2	316	127	10.5	782
X029	3170	14.9	2.40	63.9	315	128	10.8	779
X030	3120	15.1	2.35	63.1	319	128	10.7	789
Reference value	**3160**	**15.0**	**2.27**	**63.3**	**313**	**128**	**10.7**	**787**
SD	**46**	**0.2**	**0.091**	**0.86**	**7.5**	**1.4**	**0.19**	**8.8**
RSD, %	**1.5**	**1.6**	**4.0**	**1.3**	**2.4**	**1.1**	**1.8**	**1.1**

Table 3. ICP-OES measurements of reference filters, Series E. Randomly selected filters analysed at the National Institute of Occupational Health, Oslo

Analytical Wave-length in nm	Filter no. E013 µg	Filter no. E019 µg	Filter no. E027 µg	Filter no. E033 µg	Filter no. E038 µg	Filter no. E044 µg	Filter no. E051 µg	Filter no. E059 µg	Filter no. E070 µg	Filter no. E087 µg
Cd 214.438	33.2	34.2	33.1	33.1	34.0	34.2	34.4	34.4	33.2	33.3
Cd 226.502	33.0	33.3	32.9	33.0	32.9	32.9	33.1	32.7	32.9	33.1
Cr 205.552	166.2	166.7	167.2	166.2	166.1	166.3	166.7	166.6	166.4	166.1
Cr 206.149	166.6	166.9	167.6	166.9	166.2	166.7	166.7	167.3	167.0	166.4
Cr 357.869	164.9	165.0	166.0	165.1	166.2	165.0	164.5	165.4	165.3	164.8
Cu 224.700	99.3	100.1	99.4	99.3	98.5	98.5	98.6	98.2	99.0	99.3
Cu 324.754	101.3	101.6	102.4	101.6	100.8	101.1	101.3	101.7	101.7	100.9
Cu 327.396	101.4	101.4	101.6	101.0	100.9	101.0	100.9	101.2	101.0	100.7
Fe 234.349	336.4	337.1	337.1	334.4	335.6	335.5	335.2	336.0	336.1	335.7
Fe 238.204	336.3	337.7	339.0	336.2	335.7	336.3	336.8	337.5	337.7	335.5
Fe 259.940	336.9	337.6	338.0	335.4	336.4	336.4	336.5	336.9	336.7	336.0
Fe 239.562	335.5	336.9	338.8	336.0	335.0	335.4	336.6	338.1	337.3	335.1
Mn 257.610	94.6	94.9	95.0	95.1	94.5	94.6	94.7	94.8	94.7	94.3
Mn 260.569	94.5	94.8	95.2	95.1	94.4	94.5	94.5	94.7	94.7	94.2
Mn 294.920	95.0	95.1	95.1	95.2	94.9	94.8	04.9	94.8	94.8	94.6
Ni 231.604	37.1	37.5	37.1	37.2	37.0	37.0	37.1	36.8	37.1	37.3
Ni 341.476	36.5	36.9	36.9	36.7	36.8	36.5	36.4	36.3	36.9	36.7
Pb 220.353	32.1	32.6	32.2	32.3	32.3	32.2	31.9	31.4	32.1	32.6
Zn 202.548	314.2	315.7	315.3	315.0	313.0	313.6	315.3	314.5	314.7	314.6
Zn 206.200	316.4	317.7	317.3	316.7	315.3	315.7	317.4	316.7	316.6	316.5
Zn 213.856	311.8	312.9	314.0	312.6	312.1	311.1	311.5	312.3	312.5	311.6

Table 4. Corrected values obtained by ICP-OES and reference values, Series E .

Filter no.	Cd µg	Cr µg	Cu µg	Fe µg	Mn µg	Ni µg	Pb µg	Zn µg
E013	33.1	165.9	100.7	336.3	94.7	36.8	32.1	314.1
E019	33.7	166.2	101.1	337.3	94.9	37.2	32.6	315.4
E027	33.0	166.9	101.1	338.2	95.1	37.0	32.2	315.5
E033	33.1	166.1	100.6	335.5	95.1	36.9	32.3	314.8
E038	33.5	166.2	100.1	335.7	94.6	36.9	32.3	313.5
E044	33.5	166.0	100.2	335.9	94.6	36.7	32.2	313.4
E051	33.8	166.0	100.3	336.3	94.7	36.8	31.9	314.7
E059	33.5	166.4	100.4	337.1	94.8	36.5	31.4	314.5
E070	33.0	166.2	100.6	336.9	94.7	36.9	32.1	314.6
E087	33.2	165.8	100.3	335.6	94.4	37.0	32.6	314.2
Average	**33.3**	**166.2**	**100.5**	**336.5**	**94.8**	**36.9**	**32.2**	**314.5**
SD	**0.3**	**0.3**	**0.4**	**0.9**	**0.8**	**0.2**	**0.3**	**0.7**
RSD, %	**0.9**	**0.2**	**0.4**	**0.3**	**0.8**	**0.5**	**1.0**	**0.2**
Theoretical value	**33.6**	**169.7**	**99.6**	**340.8**	**97.0**	**37.2**	**32.0**	**313.0**
Recovery (%)	**99.2**	**97.9**	**100.9**	**98.7**	**97.7**	**99.2**	**100.6**	**100.6**

Table 5. Results reported by the participating laboratories, Series E - Reference filters

No.	Laboratory	Filter no.	Cd	Cr	Cu	Fe	Mn	Ni	Pb	Zn
1	HiA	E61	34	223	102	354	98	37	35	313
		E94	36	231	104	357	101	37	35	307
2	Analytica AB	E82	31.3	163	97.3	340	94.6	34.9	28.9	285
		E100	31.5	164	97.8	343	95.6	35.2	29.6	288
3	Inst.of hygiene	E52	29.90	150.30	86.95	220.50	86.45	31.50	26.33	*
		E88	30.65	152.10	88.69	236.75	89.58	32.75	27.25	*
4	Falconbrige	E49	35.1	185	103	358	100	39.1	32.4	325
		E73	34.8	186	103	352	99.3	38.9	32.1	320
5	HSE	E71	36.7	181	107	381	106	40.1	35.3	343
		E90	36.4	180	106	381	106	40.0	34.9	342
6	HAK									
7	Kuopio	E47	36	143	102	350	104	41	34	321
		E95	35	140	101	352	103	42	33	316
8	Eurofins	E64	36.3	161	111	365	112	42.7	36.2	358
		E79	33.0	150	104	346	106	38.7	32.6	330
9	Molab	E86	30.3	156	100	336	88.8	35.0	29.9	317
		E99	31.2	157	102	344	90.1	35.9	30.2	319
10	NILU	E42	35.1	170	104	369	91.8	37.9	34.7	333
		E76	34.5	166	100	366	90.2	35.1	31.1	306
11	Oulu	E45	33.6	164	105	360	94.0	36.7	34.7	312
		E97	33.7	161	106	360	93.8	36.7	34.5	308
12	Sero	E62	33.1	167	103	332	97.4	37.6	31.9	309
		E74	33.0	169	104	333	98.5	37.3	32.2	311
13	TSSH									
14	Tinfos	E65	33	173	100	305	96	35	31	288
		E93	33	175	101	310	95	35	31	288
15	X-lab	E43	31.6	164.2	93.4	334	96.7	35.8	30.8	314
		E77	31.9	164.2	93.4	334	92.1	35.8	30.8	316
16	Ørebro	E34	33.0	166	103	328	97.5	37.4	31.8	395
		E69	34.1	166	103	322	98.0	38.7	32.8	293

* Not reported

Table 6. Corrected values reported by the participating laboratories, Series E - Reference filters

No.	Laboratory	Filter no.	Cd	Cr	Cu	Fe	Mn	Ni	Pb	Zn
1	HiA	E61	34.0	223[#]	102	354	98	37.0	35.0	313
		E94	35.9	230[#]	104	356	101	36.9	34.9	306
2	Analytica AB	E82	31.3	163	97	340	95	34.9	28.9	285
		E100	31.5	164	98	343	96	35.2	29.6	288
3	Inst.of hygiene	E52	29.9	150	87	220[#]	86	31.5	26.3	
		E88	30.5	151	88	236[#]	89	32.6	27.1	
4	Falconbrige	E49	35.0	185	103	357	100	39.0	32.3	324
		E73	34.7	186	103	351	99	38.8	32.1	320
5	HSE	E71	36.6	181	107	380	106	40.0	35.2	342
		E90	36.3	180	106	380	106	39.9	34.9	342
6	HAK									
7	Kuopio	E47	36.0	143	102	350	104	40.9	34.0	321
		E95	34.9	140	101	351	103	41.9	32.9	315
8	Eurofins	E64	36.2	161	111	364	112	42.6	36.1	357
		E79	32.9	150	104	345	106	38.6	32.5	329
9	Molab	E86	30.3	156	100	336	89	35.0	29.9	317
		E99	31.1	157	102	343	90	35.8	30.1	318
10	NILU	E42	35.1	170	104	369	92	37.9	34.7	333
		E76	34.4	165	100	365	90	35.0	31.0	305
11	Oulu	E45	33.6	164	105	360	94	36.7	34.7	312
		E97	33.7	161	106	360	94	36.7	34.5	308
12	Sero	E62	33.0	166	103	331	97	37.5	31.8	308
		E74	32.9	169	104	332	98	37.2	32.1	310
13	TSSH									
14	Tinfos	E65	32.9	173	100	304	96	34.9	30.9	287
		E93	32.9	174	101	309	95	34.9	30.9	287
15	X-lab	E43	31.4	163	93	332	96	35.6	30.6	312
		E77	31.8	164	93	333	92	35.7	30.7	315
16	Ørebro	E34	33.1	166	103	329	98	37.5	31.9	396[#]
		E69	34.0	166	103	321	98	38.6	32.7	292
	Reference value		*33.6*	*170*	*99.6*	*341*	*97.0*	*37.2*	*32.0*	*313*
	X		33.4	164	101	346	97.0	37.1	32.1	314
	SD		1.95	11.7	5.30	19.2	6.06	2.60	2.48	18.1
	RSD		5.8	7.1	5.3	5.6	6.2	7.0	7.7	5.8

[#] Outlier, result rejected after applying Grubb's test method for evaluation of extreme analytical results. A signifciance level of 95 % was used.

Table 7. Results reported by the participating laboratories, Series X - Weldning fume filters

No.	Laboratory	Filter no.	Total mass	Ag	Cd	Cr	Fe	Mn	Ni	Pb
1	HiA	X036	3240	*	2.6	60	327	141	11	1650
		X059	3110	*	2.5	57	312	132	10	1700
2	Analytica AB	X012	3070	*	1.98	65.0	331	117	9.34	804
		X089	3100	*	1.82	64.9	333	117	8.74	797
3	Inst.of hygiene	X021	3046.67	*	1.82	58.20	201.38	95.43	8.35	580.89
		X069	3056.67	*	1.99	59.10	199.75	97.05	8.08	595.34
4	Falconbrige	X047	2940	15.5	1.85	70.3	339	133	12.0	813
		X097	3110	16.0	2.00	71.0	342	134	12.1	822
5	HSE	X009	26900***	*	2.3	72.8	368	141	12.1	627
		X045	25500***	*	2.2	71.2	353	138	11.7	791
6	HAK	X003	3000	*	*	*	*	*	*	*
		X056	3100	*	*	*	*	*	*	*
7	Kuopio	X019	2930	*	2	68	312	134	11	753
		X082	3110	*	2	70	332	140	12	773
8	Eurofins	X032	3110	13.2	2.23	61.2	315	129	11.2	781
		X075	3060	3.94	2.12	65.3	326	133	10.9	718
9	Molab	X016	3100	*	1.8	59.7	304	116	10.2	766
		X062	3200	*	2.0	62.2	316	119	10.6	814
10	NILU	X015	3061	2.77	2.22	74.3	345	126.9	10.8	828
		X104	3191	1.57	2.21	72.8	386	131.3	12.0	846
11	Oulu	X038	3300	*	2.20	70.0	349	130	11.3	897
		X051	3100	*	2.13	68.1	342	128	11.4	891
12	Sero	X034	**	14.6	2.01	65.5	308	127	10.0	803
		X037	**	14.7	2.01	65.2	313	127	10.3	806
13	TSSH	X025	**	*	*	*	*	*	*	*
		X076	3120	*	*	*	*	*	*	*
14	Tinfos	X023	3000	14.5	2	60	285	125	12	720
		X091	3100	15	2	62	290	127	12	740
15	X-lab	X035	**	**	**	**	**	**	**	**
		X093	3180	*	2.3	65.3	316	126.7	11.6	777
16	Ørebro	X049	3140	*	1.49	66.6	284	128	9.79	729
		X100	2880	*	1.54	64.6	283	126	9.47	720

* Not reported
** Filter cassettes with cracked middle section
*** Filter cassettes returned for exposure to welding fume without filters. A new set of filters was mounted before exposure. Results not included.

Table 8. Corrected values reported by the participating laboratories, Series X - Welding fume filters

No.	Laboratory	Filter no.	Total mass	Ag	Cd	Cr	Fe	Mn	Ni	Pb
1	HiA	X036	3270		2.62	60.6	330	142	11.1	1665[#]
		X059	3243		2.61	59.4	325	138	10.4	1773[#]
2	Analytica AB	X012	3232		2.08	68.4	348	123	9.8	846
		X089	3172		1.86	66.4	341	120	8.9	816
3	Inst.of hygiene	X021	3302		1.97	63.1	218[#]	103[#]	9.0	630
		X069	3099		2.02	59.9	203[#]	98[#]	8.2	604
4	Falconbrige	X047	3008	15.9	1.89	71.9	347	136	12.3	832
		X097	3096	15.9	1.99	70.7	340	133	12.0	818
5	HSE	X009			2.36	74.8	378	145	12.4	645
		X045			2.25	72.9	361	141	12.0	809
6	HAK	X003	3143							
		X056	3187							
7	Kuopio	X019	3099		2.12	71.9	330	142	11.6	796
		X082	3182		2.05	71.6	340	143	12.3	791
8	Eurofins	X032	3305	14.0	2.37	65.0	335	137	11.9	830
		X075	3252	4.2[#]	2.25	69.4	346	141	11.6	763
9	Molab	X016	3327		1.93	64.1	326	124	10.9	822
		X062	3229		2.02	62.8	319	120	10.7	821
10	NILU	X015	3238	2.9[#]	2.35	78.6	365	134	11.4	876
		X104	3296	1.6[#]	2.28	75.2	399	136	12.4	874
11	Oulu	X038	3346		2.23	71.0	354	132	11.5	909
		X051	3172		2.18	69.7	350	131	11.7	912
12	Sero	X034		15.3	2.11	68.6	323	133	10.5	841
		X037		15.2	2.08	67.3	323	131	10.6	832
13	TSSH	X025								
		X076	3238							
14	Tinfos	X023	3204	15.5	2.14	64.1	304	133	12.8	769
		X091	3143	15.2	2.03	62.9	294	129	12.2	750
15	X-lab	X035								
		X093	3239		2.34	66.5	322	129	11.8	791
16	Ørebro	X049	3274		1.55	69.4	296	133	10.2	760
		X100	2961		1.58	66.4	291	130	9.7	740
	Reference value		3160	*15.0*	*2.23*	*63.3*	*313*	*128*	*11.4*	*787*
	X		3202	15.3	2.12	67.9	336	134	11.1	795
	SD		94.38	0.630	0.250	4.89	25.5	6.97	1.19	75.1
	RSD		2.9	4.1	11.8	7.2	7.6	5.2	10.7	9.4

[#] Outliers, result rejected after applying Grubb's test method for evaluation of extreme analytical results. A significance level of 95 % was used. Not included in the calculations of average, SD and RSD.

Table 9. Calculated values of limits and requirements for Series E, reference filters.

Analyte	Ref. Value	OEL	% of OEL	Limit 1 %	Limit 2 %	RSD	2 RSD	Limit 1 µg	Limit 2 µg	Req.1+ µg	Req. 1- µg	Req. 2+ µg	Req. 2- µg
Cd	**33.6**	20	168	4.1	8.2	0.9	1.8	1.98	3.36	**35.6**	**31.6**	**37.0**	**30.2**
Cr	**170**	500	34	7.2	14.4	0.2	0.4	12.92	25.16	**182.6**	**156.8**	**194.9**	**144.5**
Cu	**99.6**	100	100	4.9	9.9	0.4	0.8	5.70	10.61	**105.3**	**93.9**	**110.2**	**89.0**
Fe	**341**	3000	11	10.6	21.2	0.3	0.6	38.28	74.50	**379.3**	**302.7**	**415.5**	**266.5**
Mn	**97.0**	1000	10	11.2	22.5	0.8	1.6	12.45	23.35	**109.5**	**84.5**	**120.4**	**73.6**
Ni	**37.2**	50	74	5.5	10.9	0.5	1.0	2.40	4.44	**39.6**	**34.8**	**41.6**	**32.8**
Pb	**32.0**	50	64	5.8	11.5	1.0	2.0	2.48	4.33	**34.5**	**29.5**	**36.3**	**27.7**
Zn	**313**	4000	8	12.1	24.3	0.2	0.4	39.21	77.16	**352.2**	**273.8**	**390.2**	**235.8**

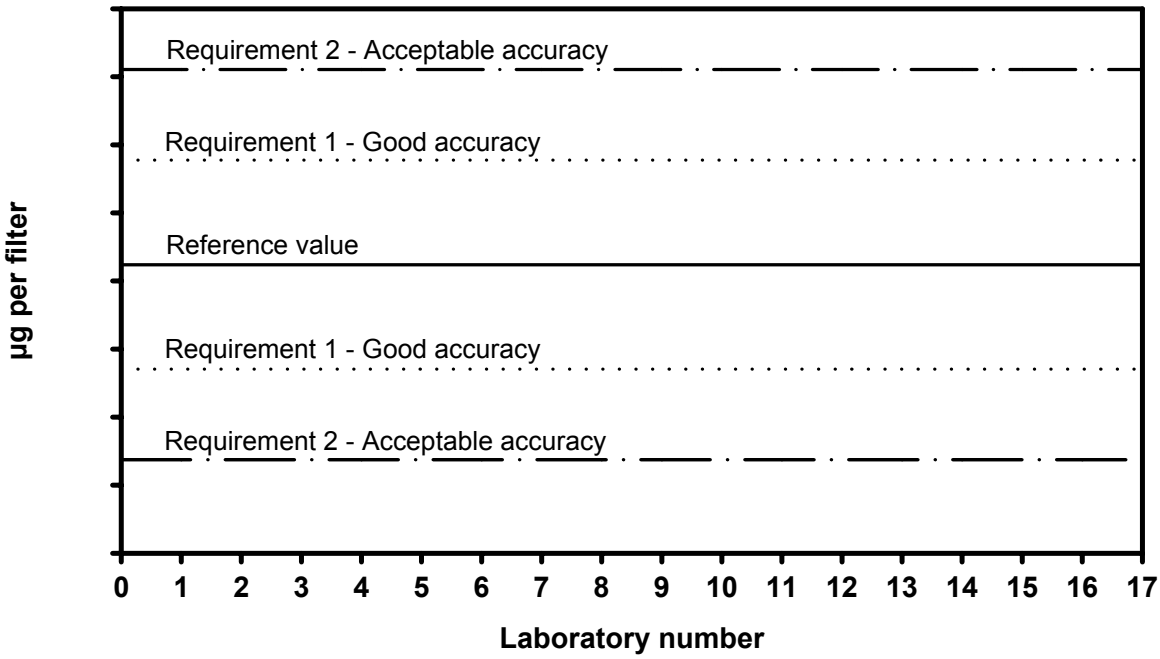
Table 10. Calculated values of limits and requirements for Series X, welding fume filters.

Analyte	Ref. Value	OEL	% of OEL	Limit 1 %	Limit 2 %	RSD	2 RSD	Limit 1 µg	Limit 2 µg	Req.1+ µg	Req.1- µg	Req.2+ µg	Req.2- µg
Tot.mass	**3160**	5000	63	5.8	11.6	1.5	3.0	277.71	460.62	**3438**	**2882**	**3621**	**2699**
Ag	**15.0**	100	15	9.6	19.3	1.6	3.2	1.92	3.37	**16.9**	**13.1**	**18.4**	**11.6**
Cd	**2.27**	20	11	10.6	21.3	4.0	8.0	0.42	0.66	**2.69**	**1.85**	**2.93**	**1.61**
Cr	**63.3**	500	13	10.2	20.5	1.3	2.6	8.12	14.59	**71.4**	**55.2**	**77.9**	**48.7**
Fe	**313**	3000	10	11.0	21.9	2.4	4.8	49.30	83.58	**362.3**	**263.7**	**396.6**	**229.4**
Mn	**128**	1000	13	10.2	20.4	1.1	2.2	15.86	28.89	**143.9**	**112.1**	**156.9**	**99.1**
Ni	**10.7**	50	21	8.5	17.0	1.8	3.6	1.29	2.20	**12.0**	**9.4**	**12.9**	**8.5**
Pb	**787**	50	1574*	4.1	9.8	1.1	2.2	56.04	94.76	**843.0**	**731.0**	**881.8**	**692.2**

* For calculations for limit 1 and 2 100 % of OEL is used (See chapter 7. Assessment criteria) .

APPENDIX 2

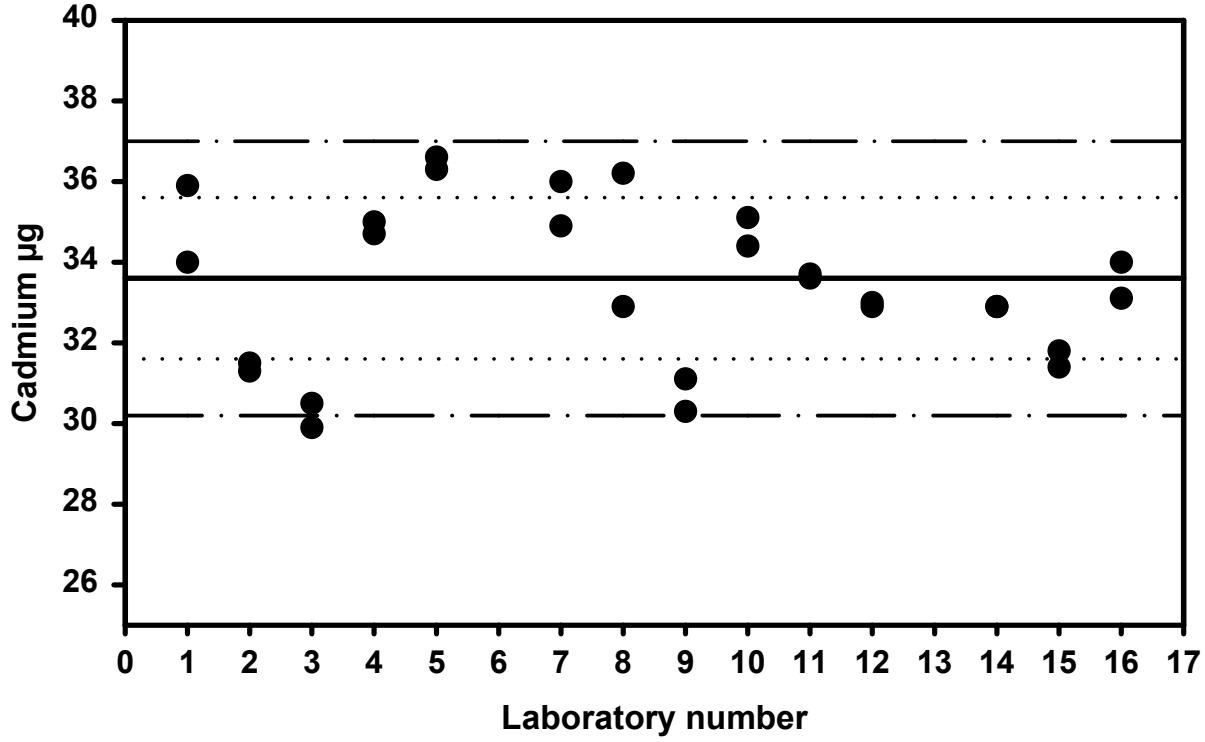
The following figure is used to illustrate the reported values from each laboratory.
The solid line represents the reference value, while the dotted lines indicate the requirements for «good» and «acceptable» results.



Cadmium - Series E

Reference value: 33.6 µg

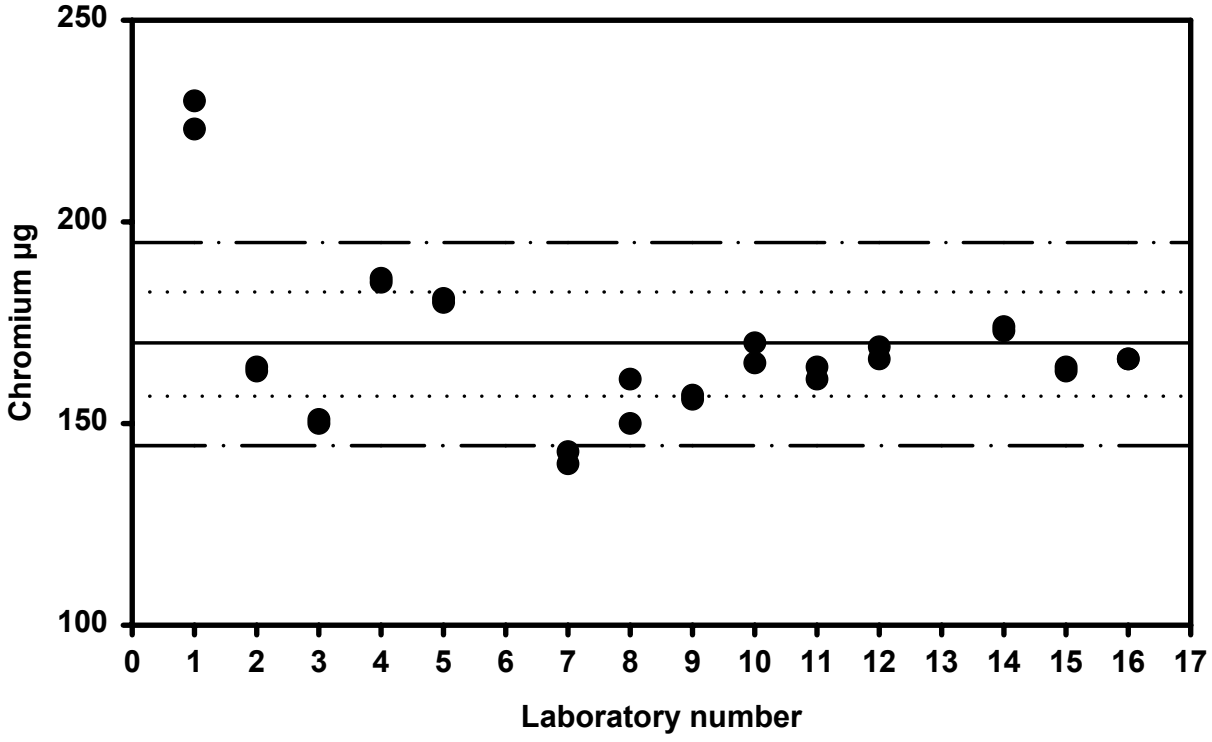
Laboratory average: 33.4 µg



Chromium - Series E

Reference value: 170 µg

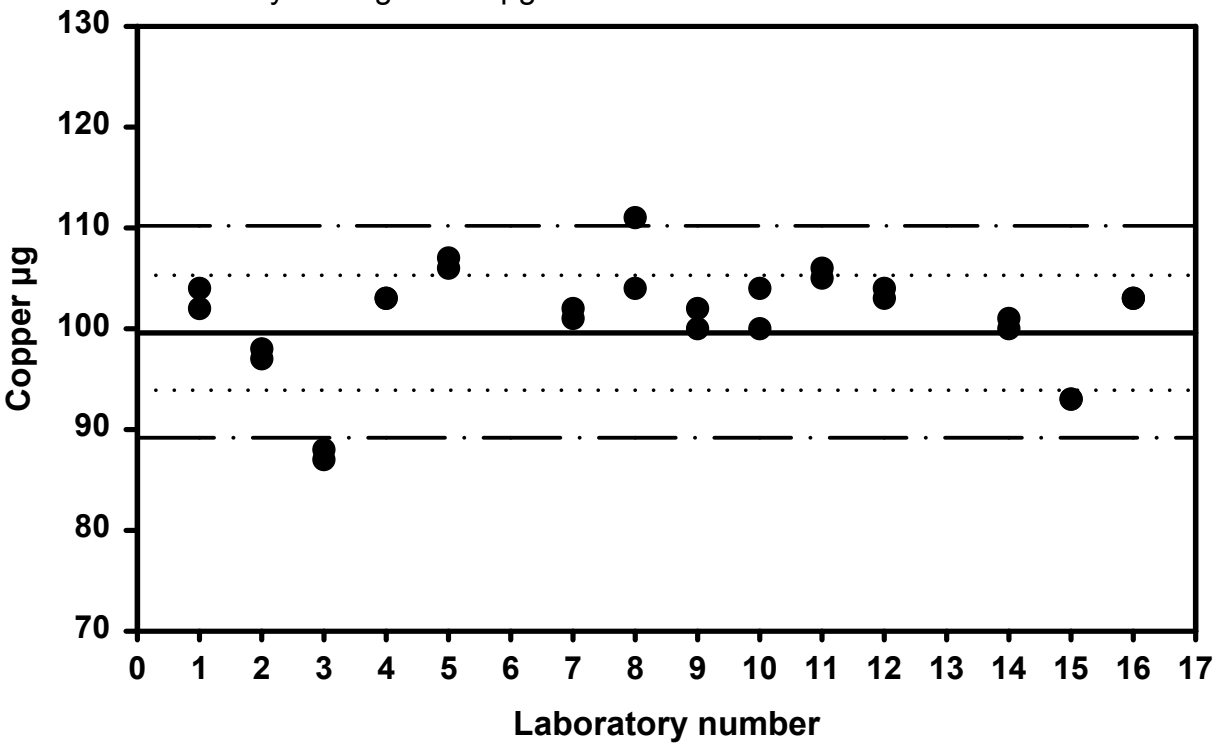
Laboratory average: 164 µg



Copper - Series E

Reference value: 99.6 µg

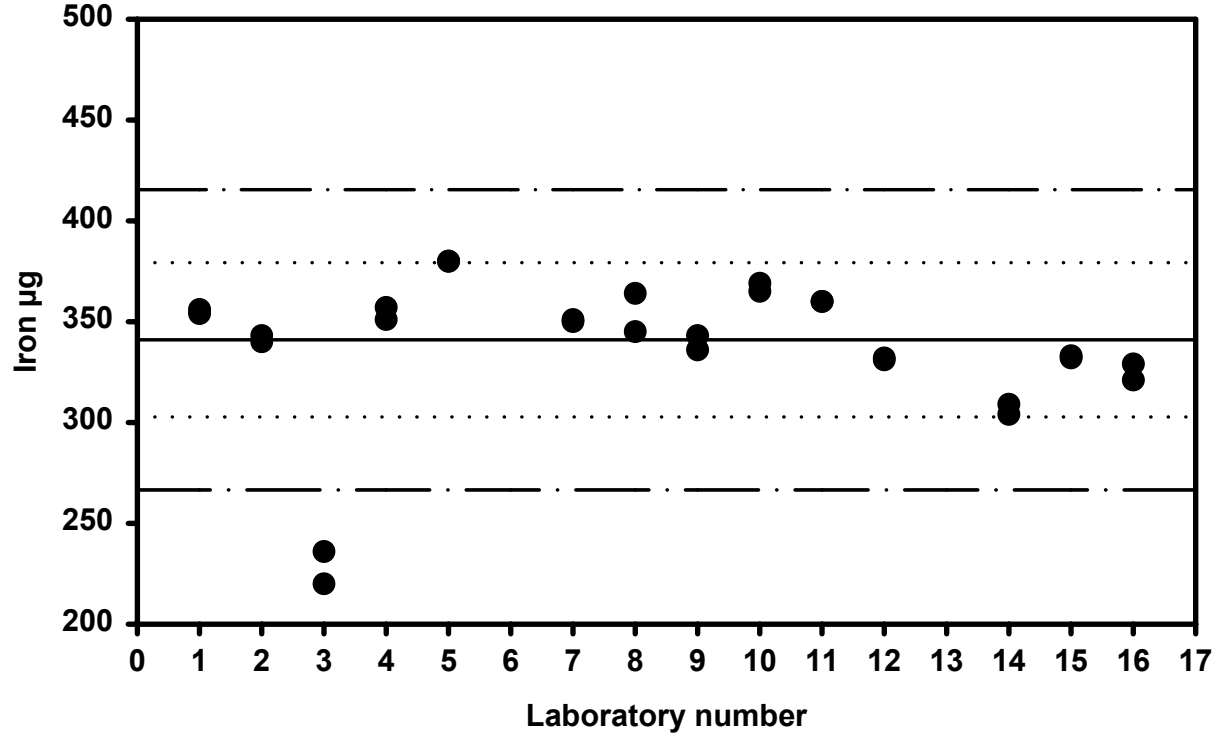
Laboratory average: 101 µg



Iron - Series E

Reference value: 341 µg

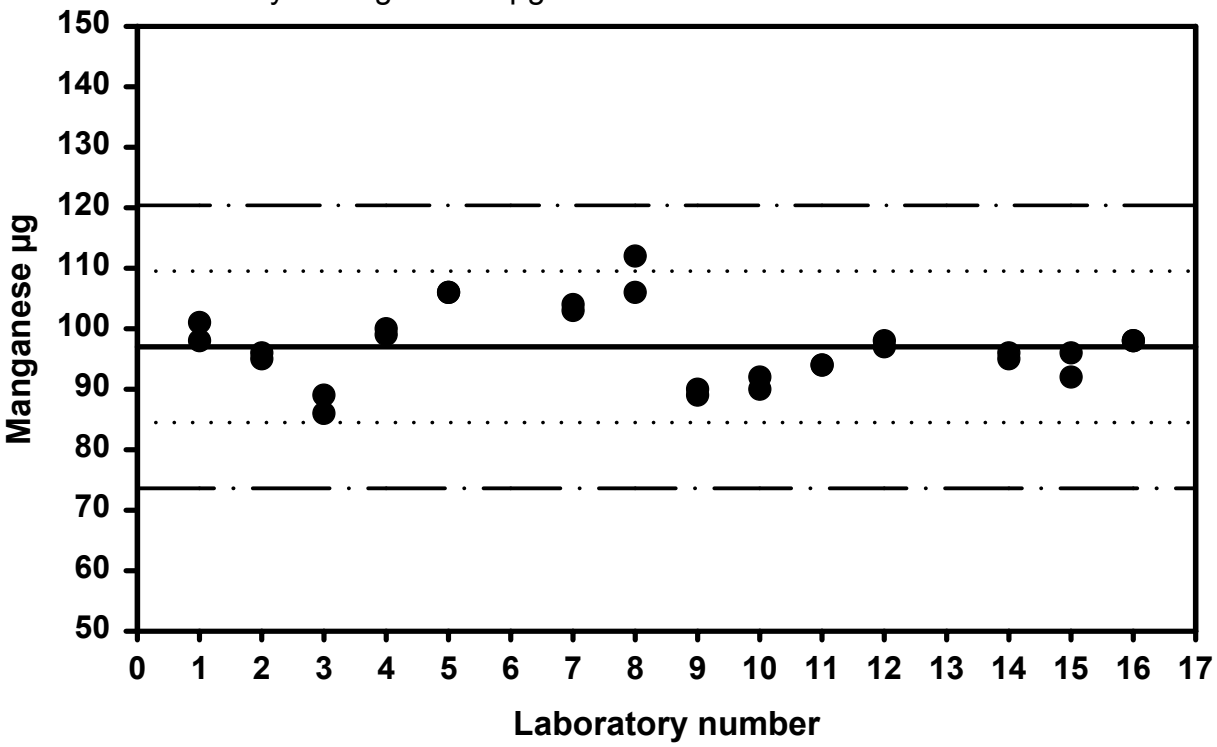
Laboratory average: 346 µg



Manganese - Series E

Reference value: 97.0 µg

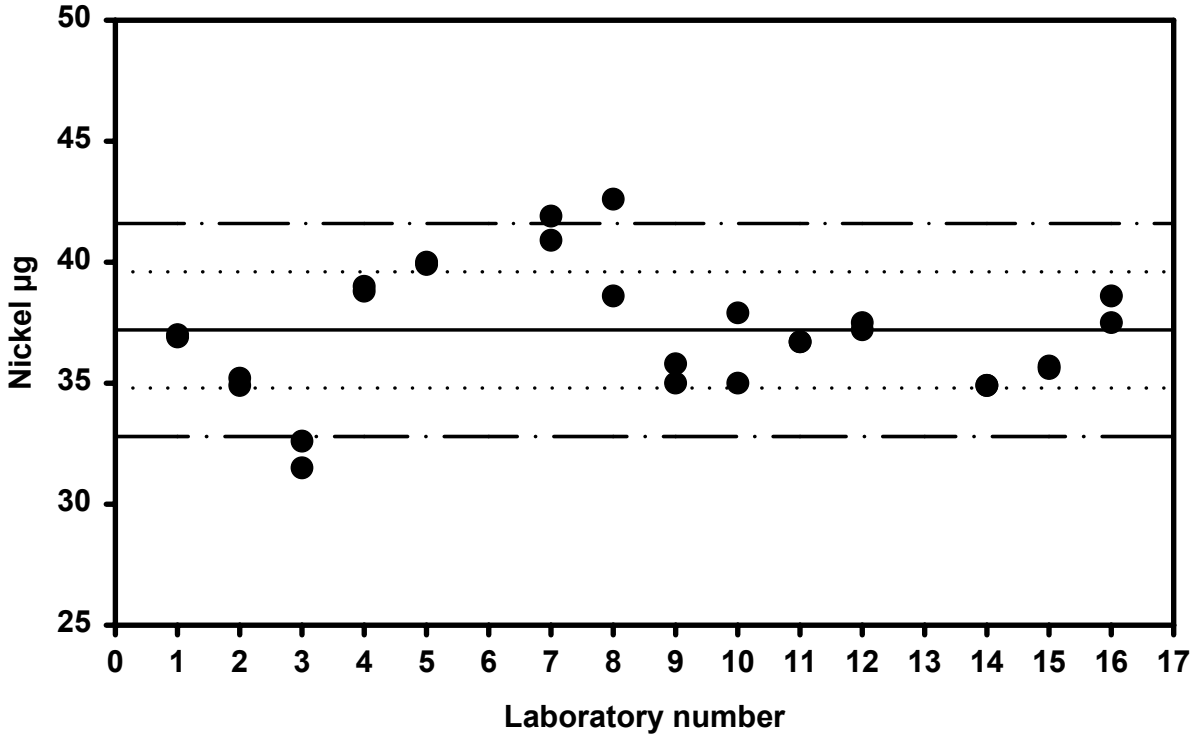
Laboratory average: 97.0 µg



Nickel - Series E

Reference value: 37.2 µg

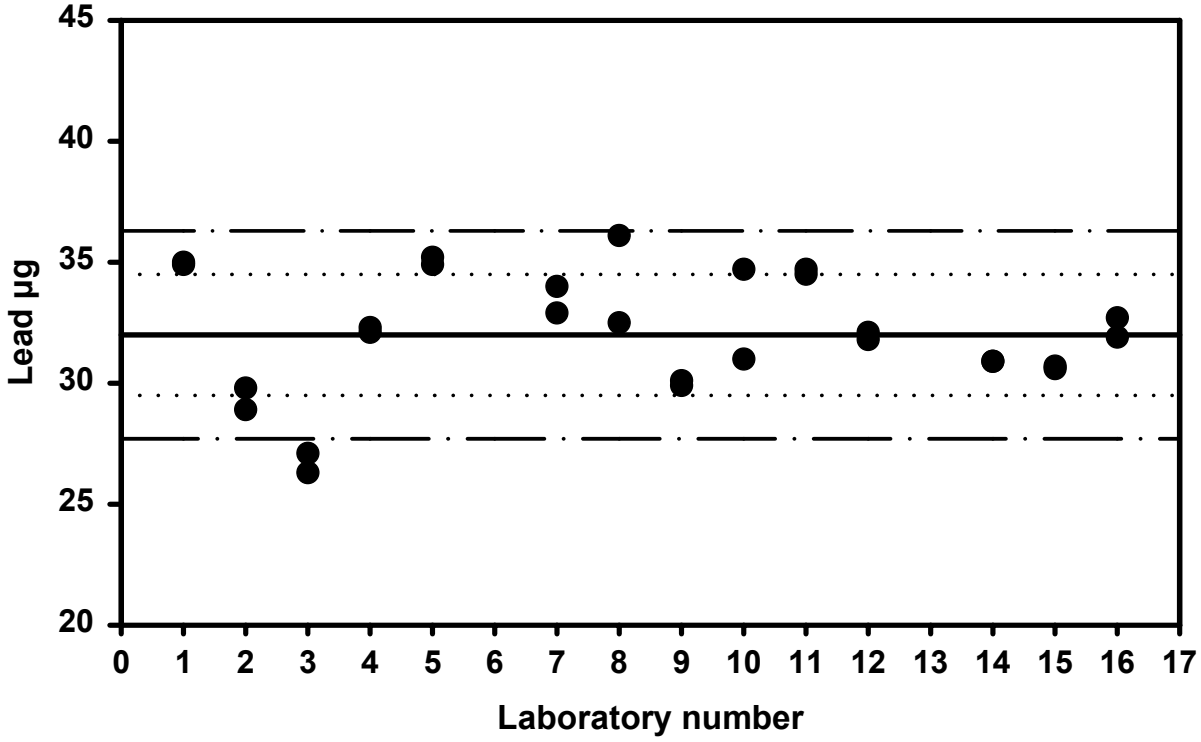
Laboratory average: 37.1 µg



Lead - Series E

Reference value: 32.0 µg

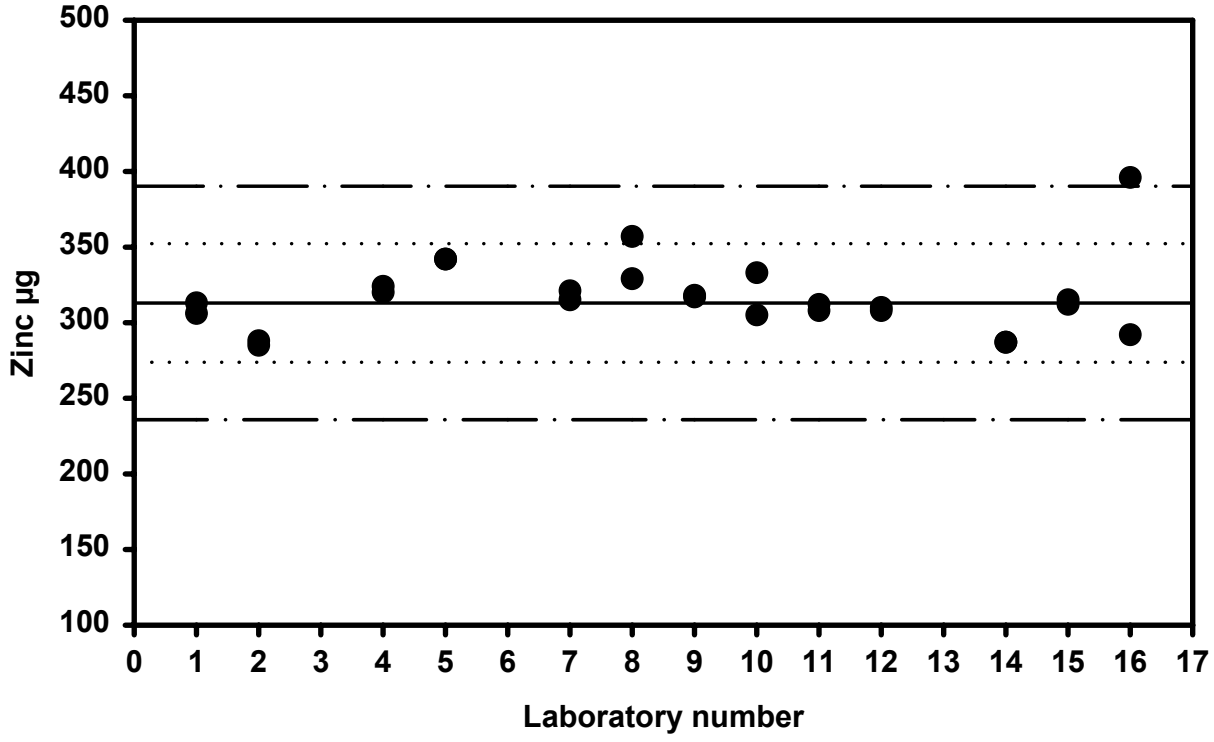
Laboratory average: 32.1 µg



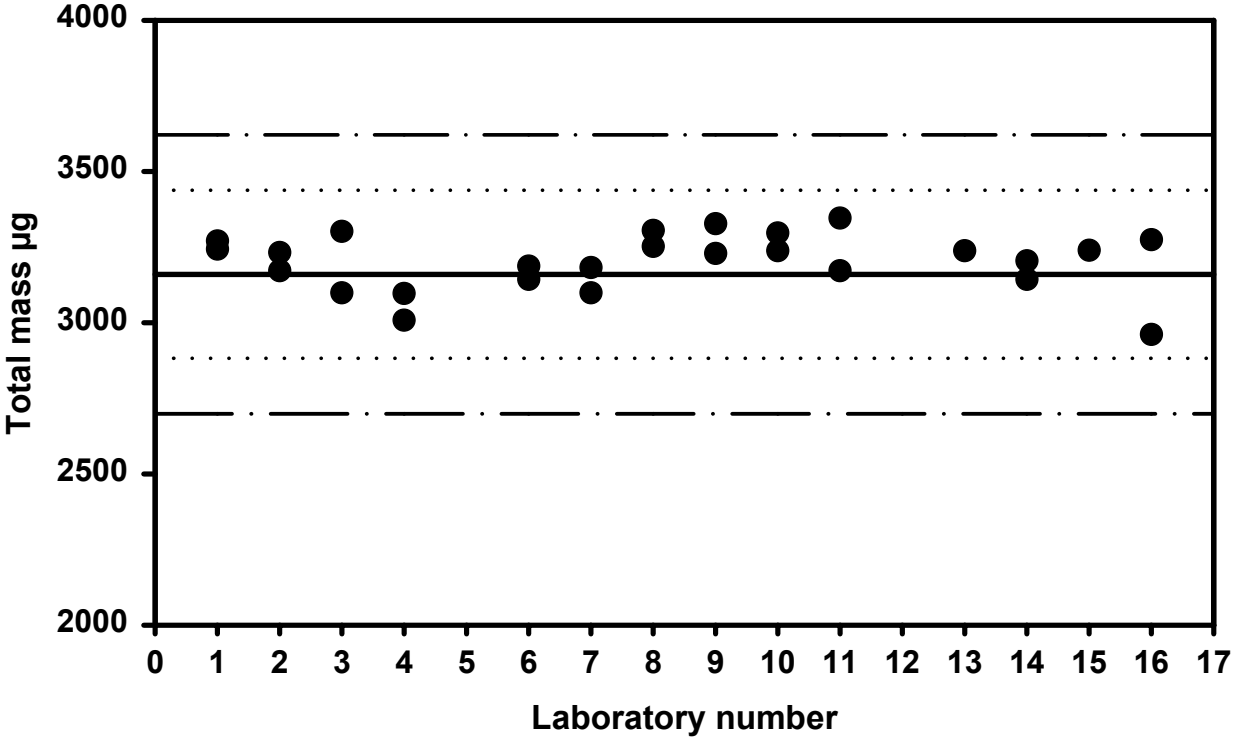
Zinc - Series E

Reference value: 313 µg

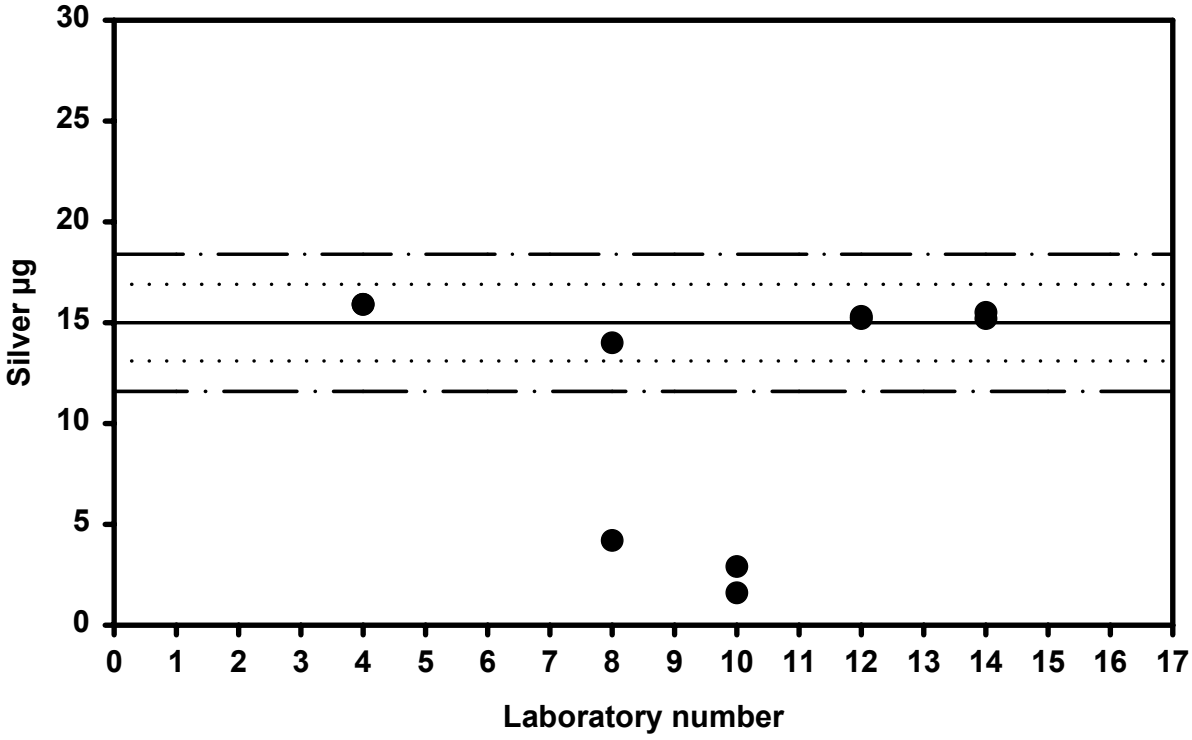
Laboratory average: 314 µg



Total mass - Series X
Reference value: 3160 µg
Laboratory average: 3202 µg



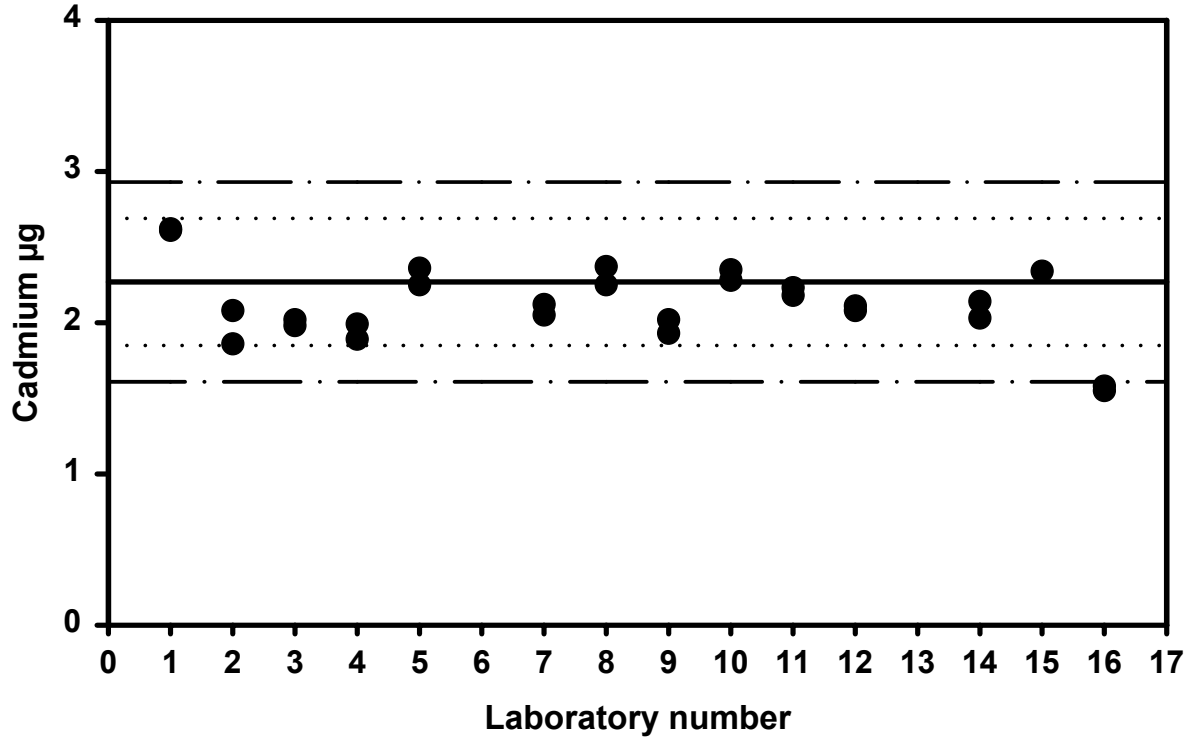
Silver - Series X
Reference value: 15.0 µg
Laboratory average: 15.3 µg



Cadmium - Series X

Reference value: 2.27 µg

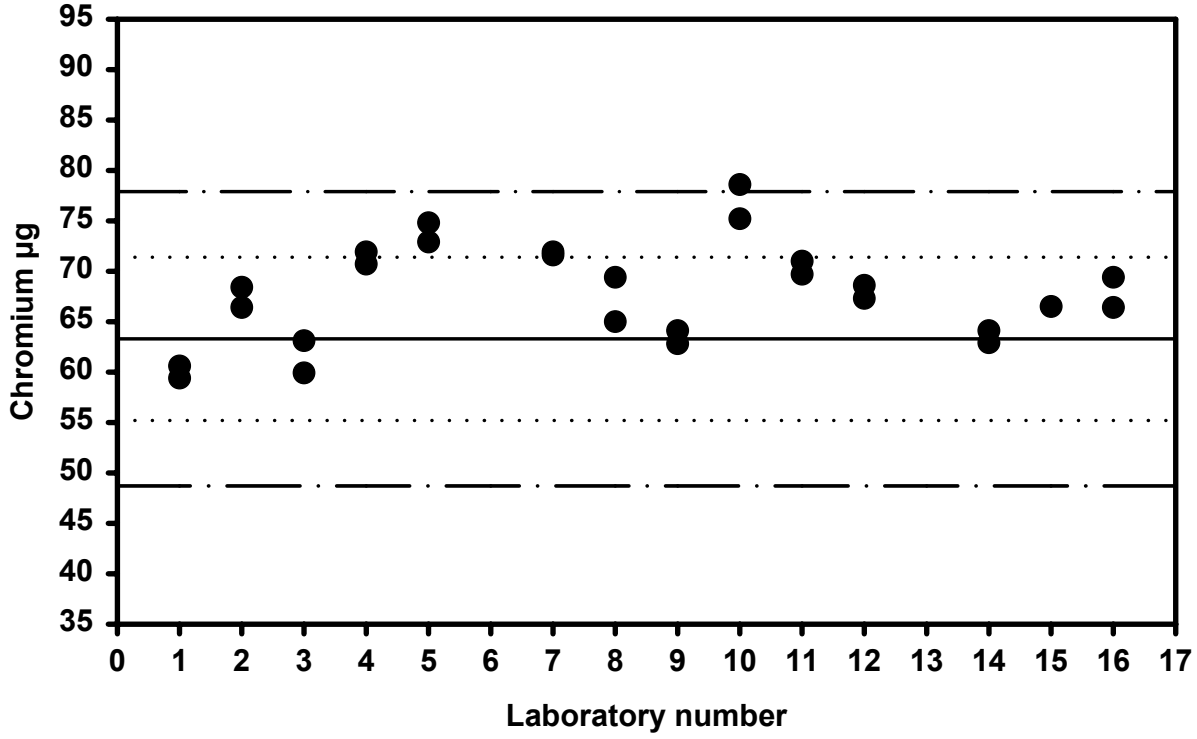
Laboratory average: 2.12 µg



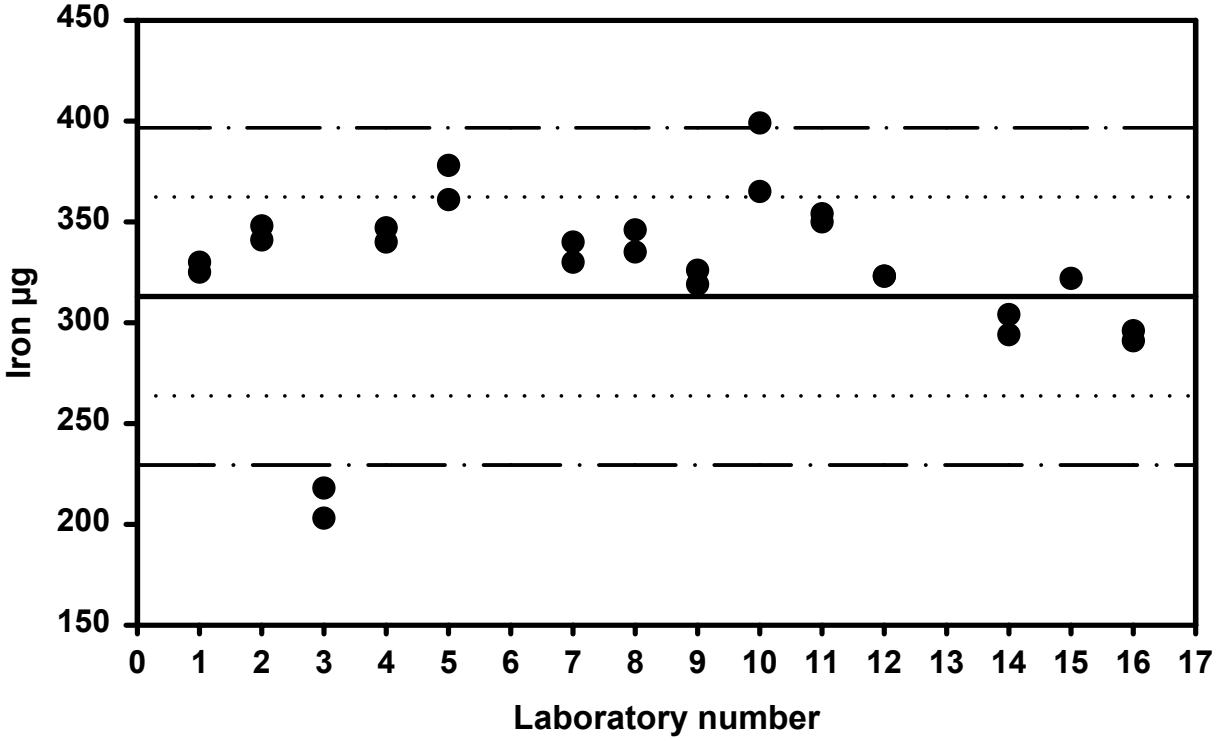
Chromium - Series X

Reference value: 63.3 µg

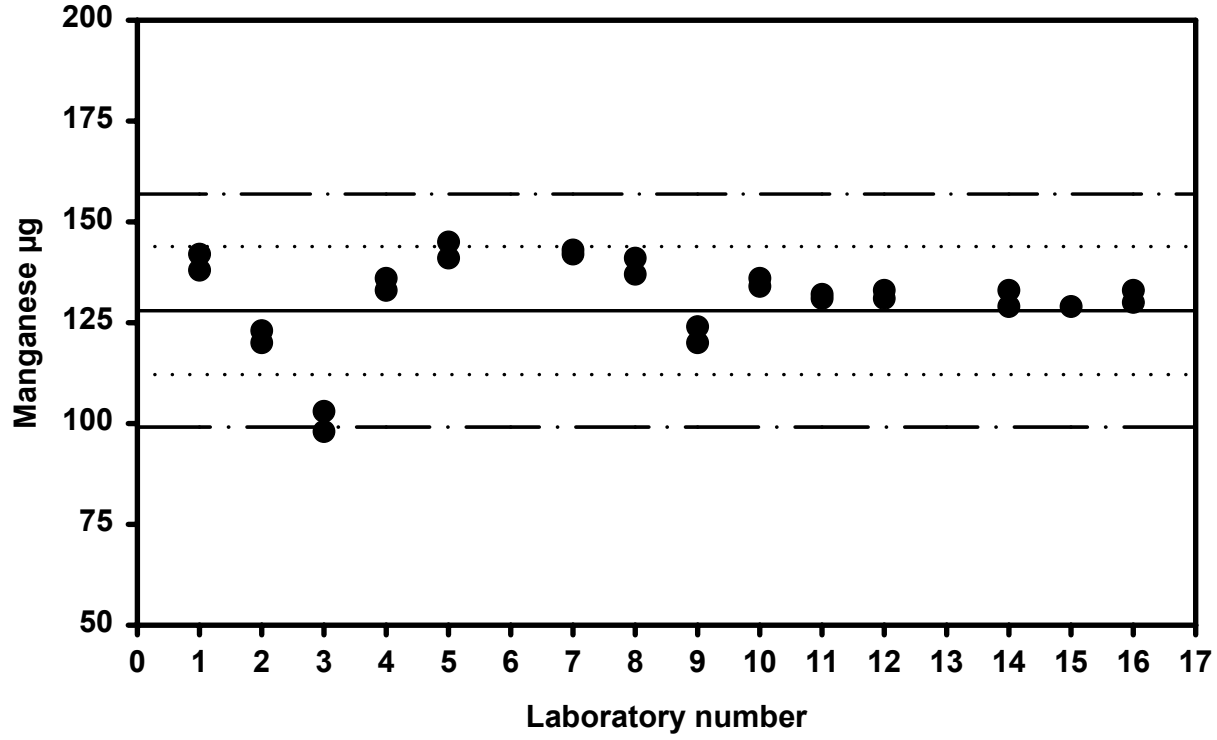
Laboratory average: 67.9 µg



Iron - Series X
Reference value: 313 µg
Laboratory average: 336 µg



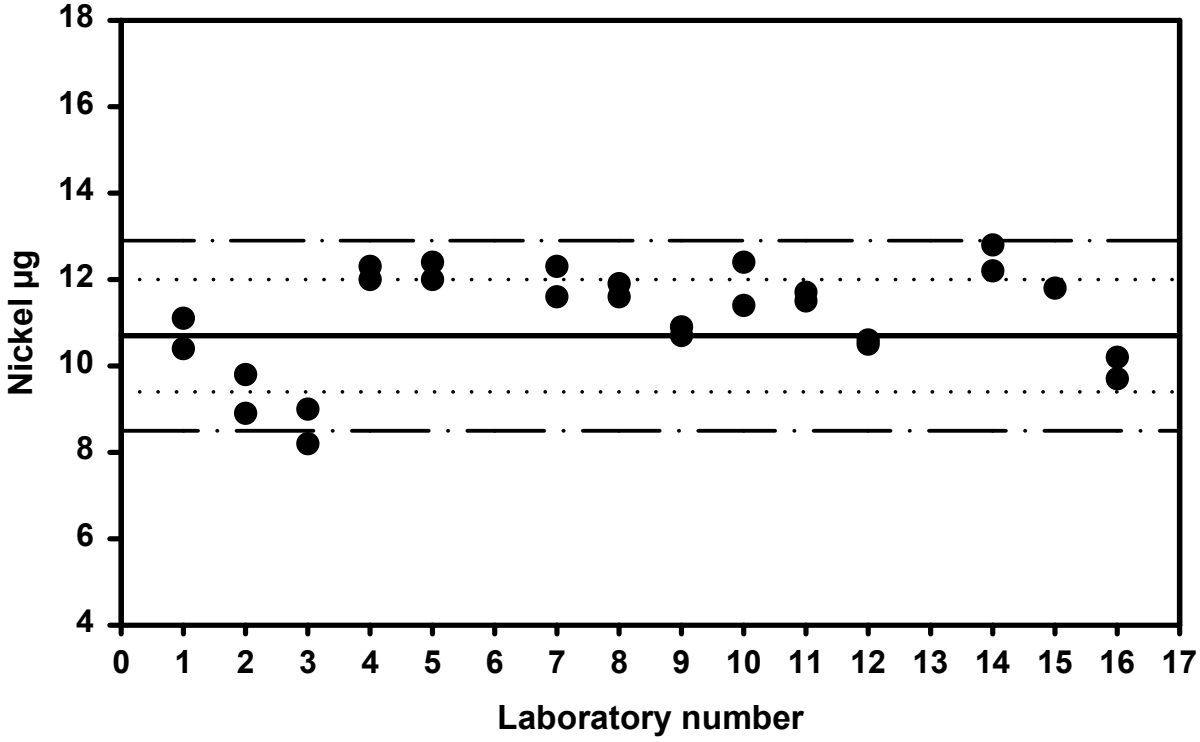
Manganese - Series X
Reference value: 128 µg
Laboratory average: 134 µg



Nickel - Series X

Reference value: 10.7 µg

Laboratory average: 11.1 µg



Lead - Series X

Reference value: 787 µg

Laboratory average: 795 µg