National Institute of Occupational Health

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

Round 11

Authors: Kari Dahl
Nils Petter Skaugset
Yngvar Thomassen

Co-ordinator: Kari Dahl

Date: 26.01.2006

Series: STAMI Report Vol. 6, No. 8 (2005) **ISSN:** 1502-0932

Summary:

In Round 11 of the proficiency-testing programme laboratories from England, Lithuania, Finland, Sweden, Denmark and Norway have participated.

Filters and cassettes were distributed to the laboratories in October 2004. The laboratories were asked to pre-weigh the filters prior to exposure to welding fume, and return the prepared filter cassettes by 19th of November 2004. Realistic workroom air filters and synthetically produced reference filters were distributed to the participating laboratories in January 2005 with a deadline of reply by 25th of February 2005.

The laboratories were asked to determine a number of occupational important elements (Ag, Cd, Cr, Fe, Mn, Ni, Pb, Zn) listed in the enclosed protocol 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 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 October 2004. The laboratories were asked to pre-weigh the filters prior to exposure to welding fume, and to return the prepared filter cassettes by 19th of November 2004. Welding fumes filters (Series Y) and synthetically produced reference filters (Series F) were distributed to the participating laboratories in January 2005 with a deadline of reply by 25th of February 2005.

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 Y (welding fume) were based on the results using ICP-OES. The reference values for Series F (reference filters) were calculated and the theoretical values verified by analysis using ICP-OES.

In this round of the proficiency- testing-programme, 17 laboratories from England, Lithuania, Finland, Sweden, Denmark and Norway have participated. Each laboratory was asked to determine a total of eight elements (Ag, Cd, Cr, Fe, Mn, Ni, Pb, Zn) in two filter matrices and total mass on the welding fume filters.

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 that 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 non-anonymously. 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 procedure.

As for the previous rounds, measurement of total mass was included in the testing programme. Filters and cassettes were distributed to the laboratories in October 2004. The laboratories were asked to pre-weigh the filters prior to exposure to welding fume, and to return the prepared filter cassettes by 19th of November 2004. The prepared samples were distributed in January 2005, with a deadline of reply by 25th of February 2005. Each participant received duplicates of workroom air filters (Series Y), reference filters spiked with known quantities of selected elements (Series F) and companion blank filters. The laboratories were asked to measure total mass (Series Y) 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, Norway	HiA
2	Analytica AB Aurorum 10, S-977 75 Luleå, Sweden	Analytica AB
3	Institute of Hygiene, Laboratory of Chemical Hazards Investigation Etmonu str. 3, LT-2001 Vilinius, Lithuania	Inst. of Hygiene
4	Kemi-laboratoriet, Danfoss A/S, Denmark	Danfoss
5	Eurofins Danmark A/S Smedeskovvej 38, DK-8464 Galten, Denmark	Eurofins
6	Falconbridge Nikkelverk A/S, Hovedlaboratoriet P.O.Box 457, N-4601 Kristiansand, Norway	Falconbridge
7	Health & Safety Executive, Inorganic Substances Section, UK	HSE
8	ITM-Luftlab, Stockholms universitet, Sweden	Stockholm
9	Kuopio Region Institut för Arbetshygien P.O.Box 93, SF-70701 Kuopio, Finland	Kuopio
10	MOLAB as P.O.Box 5000, N-8601 Mo i Rana, Norway	Molab
11	Norsk Institutt for Luftforskning Postboks 100, N-2027 Kjeller, Norway	NILU
12	Oulun Aluetyöterveyslaitos, Kemian Laboratorio Aapistie 1, SF-90220 Oulu, Finland	Oulu
13	Sero AS Postboks 24, N-1374 Billingstad, Norway	Sero
14	Telemark Sentralsjukhus, Yrkesmedisinsk avdeling Ulefossveien, 3710 Skien, Norway	TSSH
15	West Lab Services AS, Norway	West Lab
16	X-lab, Postboks 23 Ytre Laksevåg, 5848 Bergen, Norway	X-lab
17	Universitetssjukhuset Örebro, Yrkes-och miljömedicinska kliniken, Analyslaboratoriet, S-701 85 Örebro, Sweden	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 (Millipore art.no M000025A0) were used. Particulate matter was collected on 0.8 µm mixed cellulose esters membrane filters (Millipore art.no. AAWP002500).

To ensure constant rate of the air flow 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. The sampling air volume for each filter is used to calculate a correction factor to recalculate the results to a standard sampling airflow rate of 2 l/min.

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

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 4 ml HNO_3	50 ml	NS4780+NS4781 FAAS Varian Spectra AA 220 Mettler AE 240
2	Analytica AB	HNO_3/H_2O_2(reference filters)/ HNO_3 /HCl/HF(welding fume) in Teflon autoclave with microwave assisted digestion.		ICP-OES EPA 200.7 Mod ICP-SFMS EPA 200.8
3	Inst. of Hygiene	HNO_3/HCl, Teflon autoclave with microwave assisted digestion		EAAS with Zeeman and chem.mod. Balance SCALTEC SBC 21
4	Danfoss A/S	6 ml 65% HNO_3on hot plate for 1.5 hour. 0.5 ml residue diluted to volum. (Method AMIL13)		ICP-OES(Cd, Fe, Mn, Zn) EAAS (Cr, Ni) Mettler Toledo MX5
5	Eurofins	Microwave digestion using HNO_3		ICP-MS (Ag) ICP-OES (Al, Cd, Cr, Fe, Mn, Ni, Pb, Zn)
6	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	ICP-OES Varian Vista MPX Mettler 250
7	HSE	Microwave digestion using conc. HNO_3/HCl/HF		ISO15202 ICP-OES PE Optima 3000 DV ICP-OES radial mode
8	Stockholm	Only gravimetric determination		
9	Kuopio	HNO_3/HCl, Teflon autoclave with microwave assisted digestion		EAAS PE Analyst 800 (Al, Cr, Ni , Pb) FAAS PE Analyst 800 (Cd, Fe, Mn, Zn) Mettler Toledo AT 261
10	Molab	Microwave digestion using HNO_3/HCl/HF		ICP-OES NS 4860 (gravimetric determination)
11	NILU	HNO_3/H_2O_2, with microwave assisted digestion.		ICP-MS Microbalance Mettler MT-5
12	Oulu	Microwave digestion using 3 ml HNO_3 and 3ml HCl	25 ml	FAAS Perkin Elmer 5100 Mettler AT261 DeltaRange
13	Sero	HNO_3/HCl/HF digestion in Teflon autoclaves	14 ml	ICP-OES Perkin Elmer Plasma 2000
14	TSSH	Only gravimetric determination		
15	West Lab	Digestion in HNO_3 and HCl (4:1)		ICP-OES
16	X-lab	HNO_3/H_2O_2, with microwave assisted digestion		FAAS Perkin Elmer Analyst 800 Metller AT261 DeltaRange
17	Örebro	HNO_3/H_2O_2, with microwave assisted digestion. CEM MARS 5, Open wessel		ICP-QMS HP4500-100 Cahn 031 Micro Balance

6. REFERENCE VALUES

In order to determine the "true" quantities of elements and total mass on the filters, randomly selected parallel filters from each filter series were analysed at the National Institute of Occupational Health in Oslo.

Before weighing, the filters were before and after exposure conditioned min. 24 hours in a special designed laboratory at 40%±2 relative humidity and 20^0C±1. Certified reference weights (20 and 50 mg) were used to ensure the accuracy of the weighing procedure. A semi-micro-balance model Sartorius MC5 was used for the measurement of total mass.

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

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 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.
For the simultaneous measurement of all elements a Perkin-Elmer OPTIMA 3000 inductively coupled plasma optical emission spectrometer (ICP-OES) was used. Certified reference filters were used as quality control.

The reference values for Series Y (welding fumes) were 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 F) were measured by weighing. Exact values of individual filters were obtained by using a correction factor for each filter. The theoretical values were verified by results using ICP-OES measurements.

The analytical results and reference values for Series Y (welding fumes) and Series F (reference filters) are given in Appendix 1, table 1, 2, 3 and 4 respectively.

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 which is the best mathematical fit to the above measurement criteria:

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

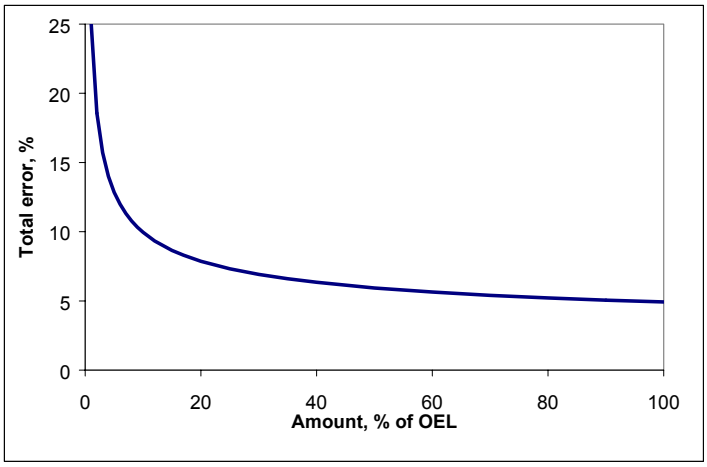


Fig. 1 Plot of the expression for limit 1

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 0.6 – 0.9 % (RSD) for Series F and between 0.7 – 1.6 % (RSD) for Series Y.

The RSD for each element and the calculations of limits and requirements for Series F and Series Y 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 reported by the participating laboratories are given in Appendix 1, Table 5 (Reference filters Series F) and Table 7 (Welding fume Series Y).

In the preparation of the spiked reference filters, the mass of the spike volume has been measured for each filter. Since the density of the reference solution was known the spiked volume for each filter could be calculated. A correction factor for the spike volume has been applied for recalculation of the reported values. The spike volumes, correction factors (spiked volume/80 µl) and the corrected values are listed in Appendix 1, Table 6.

Since the airflow during welding fume collection was different between filters on the multi-sampler, the reported results in Table 7 were recalculated to a standard sampling flow rate of 2 l/min. The air volumes, correction factors (air volume/0.180 m^3) and the corrected values are listed in Appendix 1, Table 8.

The individual results of the participating laboratories 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 F - Reference filters.

		Al	Cd	Cr	Fe	Mn	Ni	Pb	Zn
	Reference value, µg	222	14.8	47.2	514	148	59.5	36.5	223
1	HiA	●	↙	❍	❍	❍	↙		●
2	Analytica AB	●	↙	●	❍	❍	❍	↙	●
3	Inst. of Hygiene		●	❍	●	❍	↙	❍	
4	Danfoss A/S		●	●	●	●	↙	↙	●
5	Eurofins	●	●	●	●	●	●	●	●
6	Falconbridge	●	●	●	●	●	●	●	●
7	HSE	●	●	●	●	●	❍	●	●
8	Stockholm								
9	Kuopio	●	●	●	●	●	●	❍	●
10	Molab	●	●	●	●	●	●	●	●
11	NILU	❍	●	●	●	●	❍	❍	↙
12	Oulu	●	●	●	●	●	●	●	●
13	Sero	●	↙	●	●	●	●	●	●
14	TSSH								
15	West Lab	●	●	●	●	●	●	↙	●
16	X-lab		●	●	●	●	●	❍	●
17	Örebro	●	↙	●	●	●	↙	❍	❍

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

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

		Total mass	**Ag**	**Al**	**Cr**	**Fe**	**Mn**	**Ni**	**Zn**
	Reference value, µg	5247	12.5	149	43.2	538	120	21.8	21.1
1	HiA	●		❍	●	↙	↙	❍	●
2	Analytica AB	●	●	●	●	❍	●	●	●
3	Inst. of Hygiene	↙							
4	Danfoss A/S	↙		●	●	●	●	↙	●
5	Eurofins	❍	↙	●	●	●	●	●	●
6	Falconbridge	●	●	●	●	●	●	●	●
7	HSE				●	●	●	●	●
8	Stockholm	●							
9	Kuopio	●		●	●	●	●	●	●
10	Molab	❍		❍	●	●	●	●	●
11	NILU	❍		●	●	●	●	●	●
12	Oulu	❍		●	●	●	●	●	●
13	Sero	❍	❍	●	●	●	●	●	●
14	TSSH	❍							
15	West Lab	❍	●	●	●	●	●	●	●
16	X-lab	●			●	●	●	●	●
17	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 eight 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 participants were asked to mount the filters in the filter cassettes according to their routine procedure before returning them to NIOH for exposure to welding fumes. All filter cassettes that were prepared by the participating laboratories were tested according to NIOH's leak test procedure to prevent air by-pass in the filter cassettes. Filter cassettes that did not comply with the leak test requirements were pressed and re-tested together. About half of the participating laboratories returned filter cassettes that did not comply to NIOH's leak test requirements. This may be due to the fact that some of the participating laboratories do not prepare filter cassettes for air sampling as a routine procedure.

Originally the parallel sampler was designed for use with 25 mm plastic filter cassettes (Costar - Nuclepore art. no N-800932) with an extended connecting piece. These filter cassettes are no longer available. In the two previous rounds the 25 mm cassette (Millipore art. no M000025A0) with an external connecting piece and a new 25 mm cassette with an extended connecting piece (Omega Speciality Instruments Co. art. no A-002550-3) were tried. As described in two previous reports these solutions gave different problems. Before the present round of the proficiency-testing program, new external-connecting pieces for the multi-sampler unit were made and used in combination with Millipore cassettes (art. no M000025A0).

The inter-laboratory relative standard deviation after rejection of outliers, depending on the element, varies from 6.3-10.7 % for series F - reference filters and 5.0-27.7 % for series Y - welding fume filters (2.9 to 11.8 % for both filter matrices in Round 10). For all elements and both filter matrices the average standard deviation is 8.4 % (6.8 % in Round 10).

For series F – reference filters the laboratory averages show good agreement with the reference values. For series Y – welding fume filters the laboratory averages show also good agreement for Ag (few results), Cr, Mn, Ni and Zn after rejection of outliers.

The large difference between NIOH and the participating laboratories for total mass, 5247 and 4934 μg respectively, is surprising. Due to this the mass on additionally 10 cellulose ester filters and 10 PVC filters from the same welding fume series have been measured. Additionally 10 filters were posted to ensure that transportation of the filters was not the cause of the discrepancy. The results from these additional measurements only confirmed the discrepancy between the reference value of total mass and the laboratory consensus value.

A number of participants have reported difficulties in bringing the filter out of the cassette without loosing some of the welding aerosol. Few participants have also notified that some mass was deposited at the middle piece of the filter cassette. This may be due to the relatively high amount of mass collected in this round and may explain the discrepancy between the laboratory consensus value and the NIOH reference value.

Quality control filters for daily use are still 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 for elements on air filters.

Table 3 summarises the results of the proficiency-testing program since the beginning in 1990.
The distribution of results of "good accuracy", "acceptable accuracy" and "not accepted" results in round 11 is almost the same as in the last rounds.

Table 3. Summary of laboratory results for the proficiency-testing programme since the beginning in 1990

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
11	17	16	218	78	14	8	4

APPENDIX 1

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

Analytical Wave-length in nm	Filter no. Y-007 µg	Filter no. Y-015 µg	Filter no. Y-029 µg	Filter no. Y-037 µg	Filter no. Y-047 µg	Filter no. Y-061 µg	Filter no. Y-070 µg	Filter no. Y-080 µg	Filter no. Y-096 µg	Filter no. Y-104 µg
Ag328.068	12.4	12.2	12.6	12.7	12.6	12.6	12.8	12.7	12.6	12.7
Ag338.289	12.2	12.1	12.4	12.5	12.5	12.4	12.6	12.6	12.3	12.4
Al308.215	146.2	144.2	149.0	149.3	149.1	149.3	152.0	152.6	146.3	148.9
Al396.153	148.3	145.0	150.1	152.3	149.8	149.3	152.1	150.7	147.8	148.4
Cr205.560	42.6	42.2	43.5	43.5	43.5	43.4	43.8	44.2	42.4	42.8
Cr206.158	42.3	41.9	43.1	43.3	43.3	43.3	43.6	44.1	42.3	42.7
Cr357.869	42.6	42.0	43.6	43.7	43.6	43.7	44.1	44.4	42.9	43.1
Fe238.204	534.7	525.2	540.6	548.6	542.7	538.9	549.6	546.3	538.2	540.7
Fe259.939	533.6	522.9	537.8	545.4	539.6	533.7	543.7	540.2	532.9	534.3
Mn257.610	119.9	117.6	121.1	123.1	121.1	120.4	122.5	121.8	120.1	120.3
Mn260.568	119.1	117.0	120.5	122.2	120.4	119.8	121.5	121.1	119.2	119.4
Ni231.604	21.5	21.3	21.8	21.9	22.0	21.7	22.3	22.4	21.6	22.0
Ni341.476	21.4	21.2	21.8	21.9	22.0	21.7	22.2	22.2	21.3	21.7
Zn206.200	20.9	20.4	21.2	21.0	20.9	20.6	21.2	21.4	20.3	20.7
Zn202.548	21.3	20.7	21.5	21.3	21.3	21.1	21.6	21.9	20.9	21.3

Table 2. Reference values, Series Y - welding fume filters.
Data from Table 1 corrected for differences in air volume.

Filter no.	Total mass µg	Ag µg	Al µg	Cr µg	Fe µg	Mn µg	Ni µg	Zn µg
Y-007	5257	12.3	147	42.5	534	120	21.5	21.1
Y-015	5197	12.1	145	42.1	524	117	21.3	20.5
Y-029	5250	12.5	150	43.4	539	121	21.8	21.3
Y-037	5263	12.6	151	43.5	547	123	21.9	21.1
Y-047	5263	12.6	149	43.5	541	121	22.0	21.1
Y-061	5194	12.5	149	43.4	536	120	21.7	20.9
Y-070	5321	12.7	152	43.8	547	122	22.3	21.4
Y-080	5279	12.7	152	44.3	543	122	22.3	21.6
Y-096	5254	12.4	147	42.5	536	120	21.4	20.6
Y-104	5256	12.5	149	42.9	538	120	21.8	21.0
Reference value	**5253***	**12.5**	**149**	**43.2**	**538**	**120**	**21.8**	**21.1**
SD	**36.7**	**0.19**	**2.27**	**0.68**	**6.77**	**1.50**	**0.34**	**0.34**
RSD, %	**0.69***	**1.5**	**1.5**	**1.6**	**1.3**	**1.2**	**1.6**	**1.6**

* Reference values for n=20 is used for calculating requirements for Total mass in Table 10

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

Analytical Wave-length in nm	Filter no. F-50 µg	Filter no. F-76 µg	Filter no. F-77 µg	Filter no. F-78 µg	Filter no. F-79 µg	Filter no. F-80 µg	Filter no. F-81 µg	Filter no. F-82 µg	Filter no. F-84 µg	Filter no. F-85 µg
Al308.215	216	217	214	215	217	214	214	217	213	215
Al394.401	217	218	215	215	217	215	213	216	214	215
Al396.153	217	218	215	215	218	215	213	217	214	215
Cd214.440	14.2	14.0	13.8	14.2	14.1	13.9	14.0	14.1	13.9	13.9
Cd226.502	14.2	14.0	13.9	14.1	14.0	13.9	13.9	14.0	13.9	14.0
Cd228.802	14.8	14.6	14.4	14.6	14.6	14.5	14.5	14.6	14.5	14.5
Cr205.560	47.4	46.1	45.6	46.7	46.6	46.4	45.7	45.3	44.9	46.0
Cr206.158	46.2	46.8	45.6	45.9	46.7	46.4	46.2	47.0	45.9	46.3
Cr357.158	46.9	46.1	45.5	46.2	46.1	45.8	46.1	46.3	45.9	46.1
Fe234.349	515	518	508	508	515	510	506	514	509	512
Fe259.939	512	507	497	509	514	508	503	511	500	503
Fe239.562	512	516	506	508	514	508	503	510	496	502
Mn260.568	146	147	144	144	146	145	143	145	144	145
Mn294.920	146	147	145	145	147	146	144	146	144	145
Ni232.003	59.5	58.8	58.1	58.6	58.3	58.4	58.5	58.8	58.5	58.4
Ni341.476	58.9	58.0	57.5	58.2	58.2	57.6	58.1	58.0	58.1	57.9
Pb220.353	37.0	36.3	35.7	36.4	36.1	36.6	36.2	36.3	36.3	36.2
Zn206.200	225	227	223	224	225	223	221	226	223	224
Zn202.548	225	226	223	223	225	223	222	225	222	224
Zn213.857	226	226	222	223	225	223	220	223	222	224

Table 4. Corrected values obtained by ICP-OES and reference values, Series F.
Data from Table 3 corrected for differences in spiked mass.

Filter no.	Al µg	Cd µg	Cr µg	Fe µg	Mn µg	Ni µg	Pb µg	Zn µg
F-50	217	14.4	46.9	513	146	59.2	37.0	225
F-76	218	14.2	446.3	514	147	58.4	36.3	226
F-77	214	14.1	45.6	504	144	57.8	35.7	223
F-78	215	14.3	46.2	509	145	58.4	36.4	223
F-79	217	14.2	46.5	514	146	58.2	36.1	225
F-80	215	14.1	46.2	509	145	58.0	36.6	223
F-81	213	14.1	46.0	504	143	58.3	36.2	221
F-82	217	14.2	46.2	512	145	58.4	36.3	225
F-84	214	14.1	45.6	502	144	58.3	36.3	222
F-85	215	14.1	45.9	506	145	58.1	36.3	224
Average	**215**	**14.2**	**46.1**	**509**	**145**	**58.3**	**36.3**	**224**
SD	**1.5**	**0.10**	**0.40**	**4.6**	**1.1**	**0.37**	**0.33**	**1.6**
RSD, %	**0.7**	**0.7**	**0.9**	**0.9**	**0.7**	**0.6**	**0.9**	**0.7**
Theoretical value	**222**	**14.8**	**47.2**	**514**	**148**	**59.5**	**36.5**	**223**
Recovery (%)	**97.2**	**95.8**	**97.7**	**98.9**	**98.0**	**98.0**	**99.5**	**100.4**

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

No.	Laboratory	Filter no.	Al	Cd	Cr	Fe	Mn	Ni	Pb	Zn
			µg	µg	Mg	Mg	µg	µg	µg	µg
1	HiA	F-07	204	13.6	43.5	596	172	67.4	*	218
		F-35	188	12.7	40.0	603	169	69.4	*	215
2	Analytica AB	F-02	229	13.4	44.2	483	131	56.9	33.6	207
		F-37	215	12.6	41.2	453	123	52.9	31.2	194
3	Inst.of hygiene	F-12	*	14.33	52.69	563.13	163.33	73.56	39.37	*
		F-32	*	14.63	54.94	564.50	168.23	74.87	40.37	*
4	Danfoss A/S	F-03	*	14.40	48.33	508.1	148.6	22.09	49.77	220.1
		F-18	*	14.43	49.77	510.4	149.6	21.34	57.80	220.3
5	Eurofins	F-08	230	14.4	47.1	495	152	59.5	36.3	216
		F-27	230	14.3	47.0	506	154	59.2	36.2	217
6	Falconbrige	F-04	217	14.0	46.1	490	140	56.7	34.2	212
		F-23	216	14.2	46.9	492	140	57.1	35.0	215
7	HSE	F-11	220	15.4	49.4	542	157	64.4	38.1	233
		F-19	220	15.2	49.5	542	157	64.0	37.1	231
8	Stockholm	F-xx	*	*	*	*	*	*	*	*
		F-xx	*	*	*	*	*	*	*	*
9	Kuopio	F-11	227	15	49	539	148	62	32	227
		F-36	245	15	49	527	147	62	33	225
10	Molab	F-16	212	13.8	47.8	504	147	58.4	35.6	216
		F-33	229	14.0	48.6	508	148	60.8	35.8	218
11	NILU	F-09	259	15.1	47.7	552	158	60.1	40.1	23.0
		F-40	256	15.1	46.7	538	157	63.2	39.0	20.1
12	Oulu	F-14	229	15.0	48.1	527	151	59.4	36.2	225
		F-39	228	14.9	47.0	516	150	59.2	36.0	223
13	Sero	F-17	235	12.8	48.9	521	150	60.4	35.5	231
		F-38	228	13.0	47.9	519	150	60.0	33.9	226
14	TSSH	F-xx	*	*	*	*	*	*	*	*
		F-xx	*	*	*	*	*	*	*	*
15	West LAB	F-20	231.75	14.175	46.4	492.5	144.925	56.65	44.75	216.95
		F-31	236.75	14.425	42.15	497.5	144.925	59.15	37.25	221.95
16	X-lab	F-06	*	14.63	40.7	491	142.5	58.4	39.0	213
		F-28	*	14.94	40.7	497	142.5	58.4	39.0	214
17	Ørebro	F-24	228	16.0	48.2	539	148	63.7	36.8	253
		F-34	247	16.9	51.7	583	156	67.8	40.0	268

- Not reported

Table 6. Values reported by the participating laboratories corrected for differences in spiked mass, Series F - Reference filters

No.	Laboratory	Filter no.	Spiked volume, µl	Correction factor	Al µg	Cd µg	Cr µg	Fe µg	Mn µg	Ni µg	Pb µg	Zn µg
1	HiA	F-07	79.50	0.994	205	13.7	43.8	600	173	67.8	*	219
		F-35	79.62	0.995	189	12.8	40.2	606	170	69.7	*	216
2	Analytica AB	F-02	78.88	0.986	232	13.6	44.8	490	133	57.7	34.1	210
		F-37	79.40	0.993	217	12.7	41.5	456	124	53.3	31.4	195
3	Inst.of hygiene	F-12	79.38	0.992	*	14.4	53.1	568	165	74.1	39.7	*
		F-32	79.59	0.995	*	14.7	55.2	567	169	75.3	40.6	*
4	Danfoss A/S	F-03	79.29	0.991	*	14.5	48.8	513	150	22.3[#]	50.2	222
		F-18	79.41	0.993	*	14.5	50.1	514	151	21.5[#]	58.2[#]	222
5	Eurofins	F-08	79.50	0.994	231	14.5	47.4	498	153	59.9	36.5	217
		F-27	79.89	0.999	230	14.3	47.1	507	154	59.3	36.2	217
6	Falconbrige	F-04	79.05	0.988	220	14.2	46.7	496	142	57.4	34.6	215
		F-23	79.30	0.991	218	14.3	47.3	496	141	57.6	35.3	217
7	HSE	F-11	79.19	0.990	222	15.6	49.9	548	159	65.1	38.5	235
		F-19	79.33	0.992	222	15.3	49.9	547	158	64.5	37.4	233
8	Stockholm	F-xx			*	*	*	*	*	*	*	*
		F-xx			*	*	*	*	*	*	*	*
9	Kuopio	F-11	79.19	0.990	229	15.2	49.5	545	150	62.6	32.3	229
		F-36	79.61	0.995	246	15.1	49.2	530	148	62.3	33.2	226
10	Molab	F-16	79.52	0.994	213	13.9	48.1	507	148	58.8	35.8	217
		F-33	79.60	0.995	230	14.1	48.8	511	149	61.1	36.0	219
11	NILU	F-09	79.31	0.991	261	15.2	48.1	557	159	60.6	40.4	23[#]
		F-40	79.40	0.993	258	15.2	47.1	542	158	63.7	39.3	20[#]
12	Oulu	F-14	79.39	0.992	231	15.1	48.5	531	152	59.9	36.5	227
		F-39	79.41	0.993	230	15.0	47.3	520	151	59.6	36.3	225
13	Sero	F-17	79.40	0.993	237	12.9	49.3	525	151	60.9	35.8	233
		F-38	79.60	0.995	229	13.1	48.1	522	151	60.3	34.1	227
14	TSSH	F-xx			*	*	*	*	*	*	*	*
		F-xx			*	*	*	*	*	*	*	*
15	West Lab	F-20	79.42	0.993	233	14.3	46.7	496	146	57.1	45.1	219
		F-31	79.54	0.994	238	14.5	42.4	500	146	59.5	37.5	223
16	X-lab	F-06	79.39	0.992	*	14.7	41.0	495	144	58.8	39.3	215
		F-28	79.39	0.992	*	15.1	41.0	501	144	58.8	39.3	216
17	Ørebro	F-24	79.36	0.992	230	16.1	48.6	543	149	64.2	37.1	255
		F-34	79.65	0.996	248	17.0	51.9	586	157	68.1	40.2	269
	Reference value				*222*	*14.8*	*47.2*	*514*	*148*	*59.5*	*36.5*	*223*
	X				229	14.5	47.4	527	151	62.1	36.2	224
	SD				15.6	1.0	3.6	34.6	10.3	5.1	3.88	14.0
	RSD				6.8	6.6	7.5	6.6	6.8	8.2	10.7	6.3

* Not reported

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

Table 7. Results reported by the participating laboratories, Series Y - Welding fumes filters

No.	Laboratory	Filter no.	Total mass, µg	Ag µg	Al µg	Cr µg	Fe µg	Mn µg	Ni µg	Zn µg
1	HiA	Y-019	4870	*	94	38.6	555	136	23.4	22.2
		Y-087	5240	*	103	41.1	694	150	22.9	18.8
2	Analytica AB	Y-006	5.0[1]	13.1	148	44.2	508	120	22.7	21.3
		Y-079	5.1[1]	11.6	129	38.9	457	107	20.4	19.0
3	Inst.of hygiene	Y-032	3860.0	*	*	*	*	*	*	*
		Y-099	3680.0	*	*	*	*	*	*	*
4	Danfoss A/S	Y-025	50.37	*	*	46.87	517.0	122.1	62.75	19.49
		Y-058	49.81	*	*	44.79	512.7	120.0	63.68	19.38
5	Eurofins	Y-046	4890	6.2	150	42.2	498	121	20.8	21.4
		Y-101	4720	4.9	150	42.5	490	120	21.1	21.2
6	Falconbrige	Y-021	4760	11.3	123	41.6	489	110	22.0	19.2
		Y-072	4990	11.6	124	42.6	495	114	22.3	20.1
7	HSE	Y-107	2541***	*	140	46.0	543	124	23.7	23.7
		Y-039	2336***	*	137	44.8	526	121	23.0	20.8
8	Stockholm	Y-055**	4968							
		Y-081	5202							
9	Kuopio	Y-012	4810	*	157	45	547	122	22	23
		Y-069	4780	*	157	45	538	120	21	22
10	Molab	Y-043	4.95[1]	*	136	47.3	528	122	22.8	20.1
		Y-097	4.91[1]	*	110	42.4	483	113	20.9	18.4
11	NILU	Y-028	4700	*	150	42.4	501	119	20.6	20.1
		Y-062	4840	*	143	41.7	491	114	22.3	23.0
12	Oulu	Y-010	4740	*	138	40.6	506	117	20.8	19.4
		Y-056	4550	*	147	44.1	558	123	21.3	20.3
13	Sero	Y-004	4780	13.9	141	42.1	513	113	20.0	19.4
		Y-036	4690	13.7	140	41.7	497	111	20.0	18.9
14	TSSH	Y-035****	4.73[1]							
		Y-066	4.92[1]							
15	West Lab	Y-002	4.55[1]	11.425	134.25	38.65	480	112.425	19.9	20.7
		Y-065	4.57[1]	11.675	124.25	43.4	490	114.925	20.9	21.95
16	X-lab	Y-052	4870	*	*	40.7	523	120.8	23.4	21
		Y-105	5200	*	*	42.1	546	125.6	23.4	21
17	Ørebro	Y-022	5.011[1]	*	99.2	42.6	561	121	22.5	23.8
		Y-109	5.227[1]	*	96.4	41.7	542	119	21.7	23.7

* Not reported
** Filter cassette with material deposited on the middle ring reported
*** Filter cassettes not returned for exposure to welding fume. A new set of filter cassettes was mounted before exposure. Results not included.
**** Material deposited on the support pad reported.
[1] Reported in mg – recalculated to µg and included.

Table 8. Values reported by the participating laboratories corrected for differences in air volume, Series Y - Welding fume filters

No.	Laboratory	Filter no.	Airvolume, m^3	Correction factor	Total mass, µg	Ag µg	Al µg	Cr µg	Fe µg	Mn µg	Ni µg	Zn µg
1	HiA	Y-019	0.174	0.967	5038	*	97.2	39.9	574	141	24.2	23.0
		Y-087	0.180	1.000	5240	*	103	41.1	694#	150#	22.9	18.8
2	Analytica AB	Y-006	0.173	0.961	5202	13.6	154	46.0	529	125	23.6	22.2
		Y-079	0.180	1.000	5100	11.6	129	38.9	457	107	20.4	19.0
3	Inst.of hygiene	Y-032	0.173	0.961	4016	*	*	*	*	*	*	*
		Y-099	0.183	1.017	3620#	*	*	*	*	*	*	*
4	Danfoss A/S	Y-025	0.174	0.967	52#	*	*	48.5	535	126	64.9#	20.2
		Y-058	0.176	0.978	51#	*	*	45.8	524	123	65.1#	19.8
5	Eurofins	Y-046	0.174	0.967	5059	6.4	155	43.7	515	125	21.5	22.1
		Y-101	0.177	0.983	4800	5.0	152	43.2	498	122	21.5	21.6
6	Falconbrige	Y-021	0.174	0.967	4924	11.7	127	43.0	506	114	22.8	19.9
		Y-072	0.176	0.978	5103	11.9	127	43.6	506	117	22.8	20.6
7	HSE	Y-107	0.181	1.006	***	*	139	45.7	540	123	23.6	23.6
		Y-039	0.176	0.978	***	*	140	45.8	538	124	23.5	21.3
8	Stockholm	Y-055**	0.177	0.983	5052	*	*	*	*	*	*	*
		Y-081	0.180	1.000	5202	*	*	*	*	*	*	*
9	Kuopio	Y-012	0.174	0.967	4976	*	162	46.6	566	126	22.8	23.8
		Y-069	0.176	0.978	4889	*	161	46.0	550	123	21.5	22.5
10	Molab	Y-043	0.178	0.989	5006	*	138	47.8	534	123	23.1	20.3
		Y-097	0.183	1.017	4830	*	108	41.7	475	111	20.6	18.1
11	NILU	Y-028	0.175	0.972	4834	*	154	43.6	515	122	21.2	20.7
		Y-062	0.179	0.994	4867	*	144	41.9	494	115	22.4	23.1
12	Oulu	Y-010	0.171	0.950	4989	*	145	42.7	533	123	21.9	20.4
		Y-056	0.177	0.983	4627	*	150	44.8	568	125	21.7	20.6
13	Sero	Y-004	0.174	0.967	4945	14.4	146	43.6	531	117	20.7	20.1
		Y-036	0.177	0.983	4769	13.9	142	42.4	505	113	20.3	19.2
14	TSSH	Y-035****	0.178	0.989	4783	*	*	*	*	*	*	*
		Y-066	0.175	0.972	5061	*	*	*	*	*	*	*
15	West Lab	Y-002	0.174	0.967	4707	11.8	139	40.0	497	116	20.6	21.4
		Y-065	0.176	0.978	4674	11.9	127	44.4	501	118	21.4	22.4
16	X-lab	Y-052	0.179	0.994	4897	*	*	40.9	526	122	23.5	21.1
		Y-105	0.181	1.006	5171	*	*	41.9	543	125	23.3	20.9
17	Ørebro	Y-022	0.175	0.972	5154	*	102	43.8	577	125	23.1	24.5
		Y-109	0.182	1.011	5170	*	95.3	41.2	536	118	21.5	23.4
	Reference value				5247	*12.5*	*149*	*43.2*	*539*	*120*	*21.8*	*21.1*
	X				4934	11.2	138	43.5	525	121	22.2	21.2
	SD				244	3.1	18.1	2.4	29.0	6.5	1.2	1.6
	RSD				5.0	27.7	13.1	5.6	5.5	5.3	5.3	7.7

* Not reported
** Filter cassette with material deposited on the middle ring reported
*** Filter cassettes not returned for exposure to welding fume. A new set of filter cassettes was mounted before exposure. Results not included.
**** Material deposited on the support pad reported.
# 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 F, reference filters.

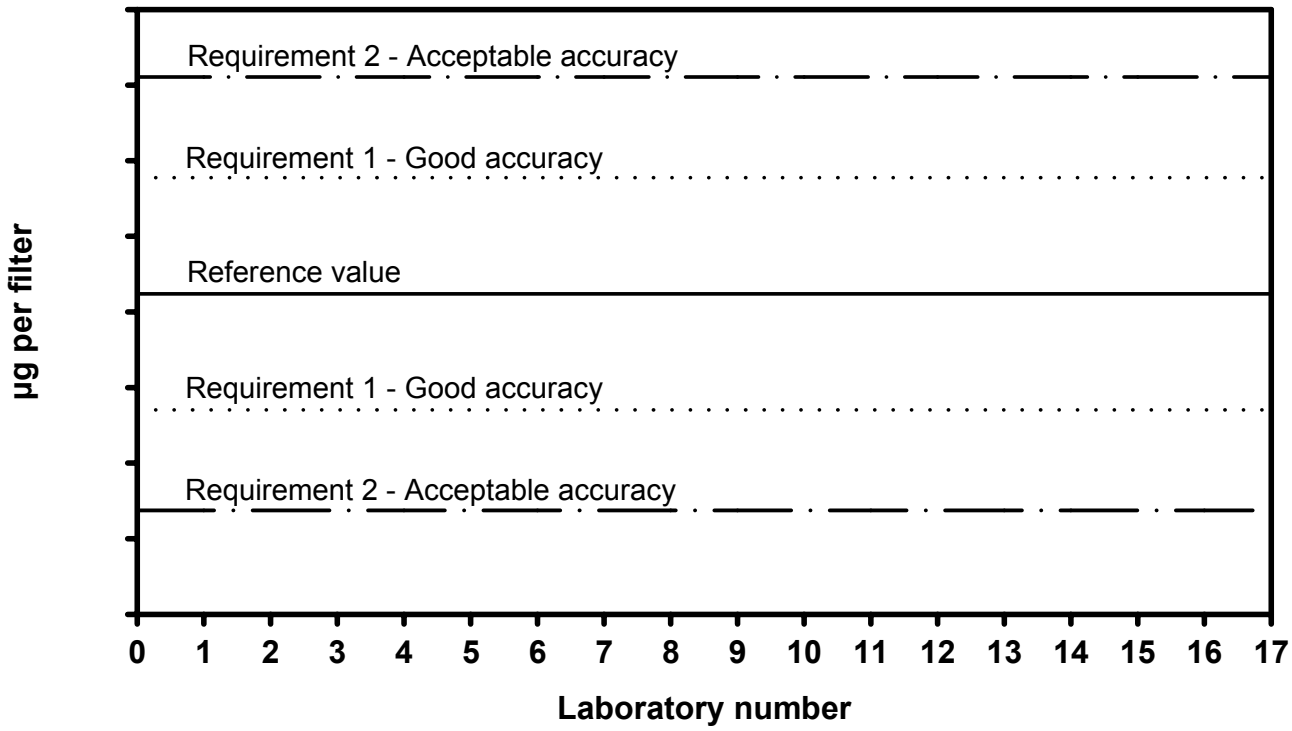
Analyte	Ref. Value	OEL	% of OEL	Limit 1 %	Limit 2 %	RSD	2 RSD	Limit 1 µg	Limit 2 µg	Req.1+ µg	Req. 1- µg	Req. 2+ µg	Req. 2- µg
Al	**222**	5000	4	14.8	29.6	0.7	1.4	36.01	68.91	**258.0**	**186.0**	**290.9**	**153.1**
Cd	**14.8**	20	74	5.5	10.9	0.7	1.4	1.02	1.83	**15.8**	**13.8**	**16.6**	**13.0**
Cr	**47.2**	500	9	11.3	22.7	0.9	1.8	6.21	11.56	**53.4**	**41.0**	**58.8**	**35.6**
Fe	**541**	3000	18	9.0	18.0	0.9	1.8	58.55	107.37	**599.6**	**482.4**	**648.4**	**433.6**
Mn	**148**	1000	15	9.7	19.4	0.7	1.4	16.39	30.72	**164.4**	**131.6**	**178.7**	**117.3**
Ni	**59.5**	50	119	4.6	9.3	0.6	1.2	3.47	6.22	**63.0**	**56.0**	**65.7**	**53.3**
Pb	**36.5**	50	73	5.5	11.0	0.9	1.8	2.66	4.67	**39.3**	**33.8**	**41.2**	**31.8**
Zn	**223**	4000	6	13.7	27.3	0.7	1.4	33.61	64.10	**256.6**	**189.4**	**287.1**	**158.9**

Table 10. Calculated values of limits and requirements for Series Y, welding fumes 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	**5247**	5000	105	4.8	9.7	1.0	2.0	358.7	612.4	**5606**	**4888**	**5859**	**4635**
Ag	**12.5**	100	13	10.3	20.5	1.5	3.0	1.02	1.83	**15.8**	**13.8**	**16.6**	**13.0**
Al	**149.4**	5000	3	17.1	34.1	1.5	3.0	6.21	11.56	**53.4**	**41.0**	**58.8**	**35.6**
Cr	**43.2**	500	9	11.7	23.4	1.6	3.2	42.37	78.60	**383.4**	**298.6**	**419.6**	**262.4**
Fe	**538**	3000	18	9.0	18.1	1.3	2.6	16.39	30.72	**164.4**	**131.6**	**178.7**	**117.3**
Mn	**120**	1000	12	10.4	20.8	1.2	2.4	3.47	6.22	**63.0**	**56.0**	**65.7**	**53.3**
Ni	**21.8**	50	44	6.6	13.2	1.6	3.2	2.66	4.67	**39.2**	**33.8**	**41.2**	**31.8**
Zn	**21.1**	4000	1	31.5	63.0	1.6	3.2	7.32	13.97	**28.4**	**13.8**	**35.1**	**7.1**

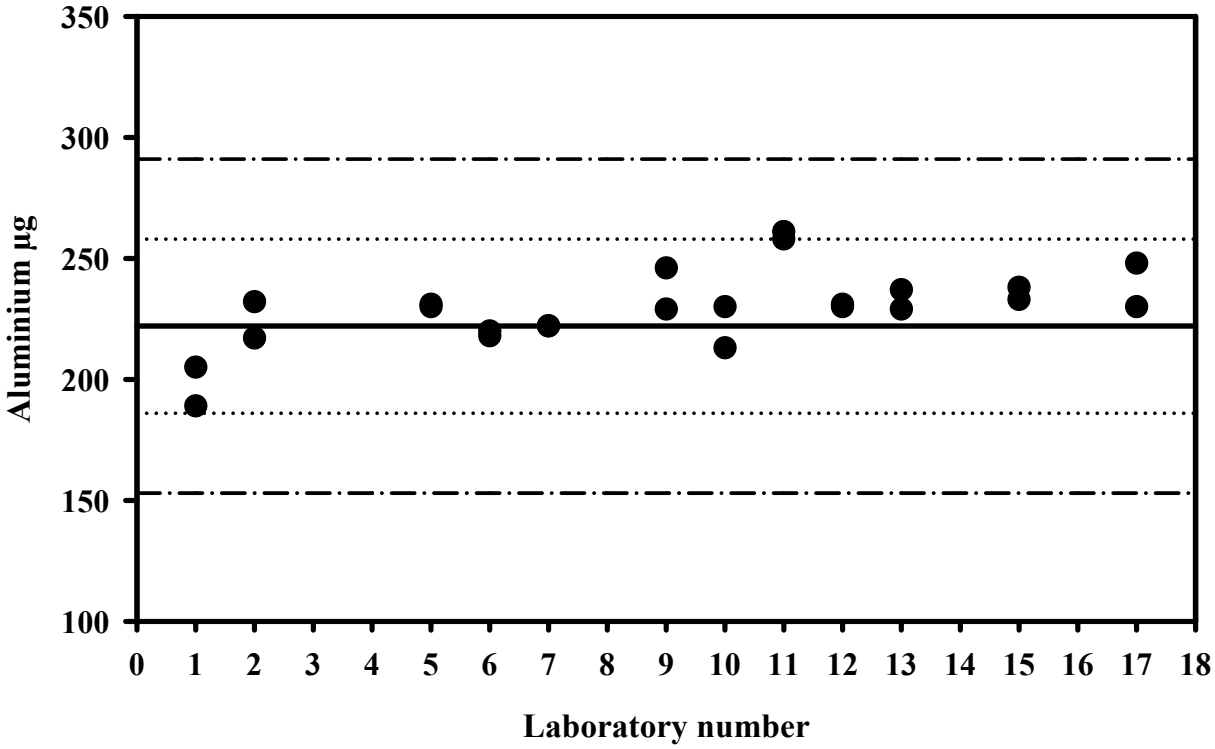
APPENDIX 2

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.



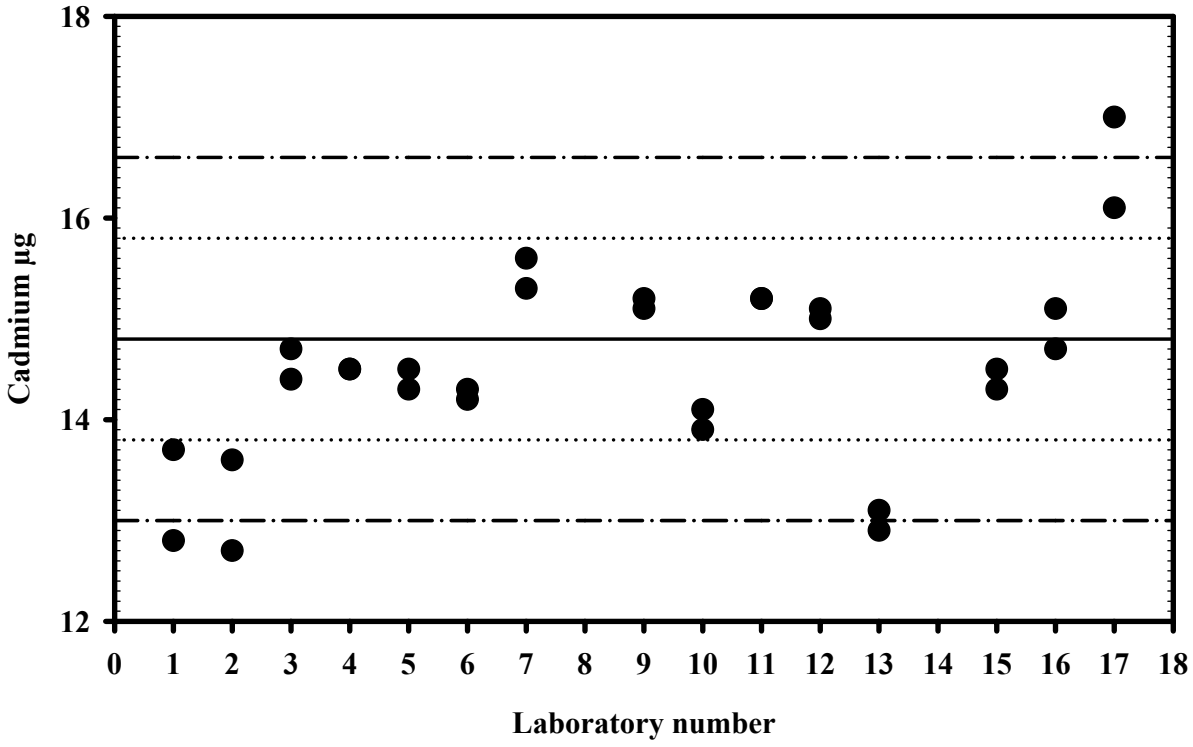
Aluminium - Series F

Reference value: 222 µg
Laboratory average: 229 µg



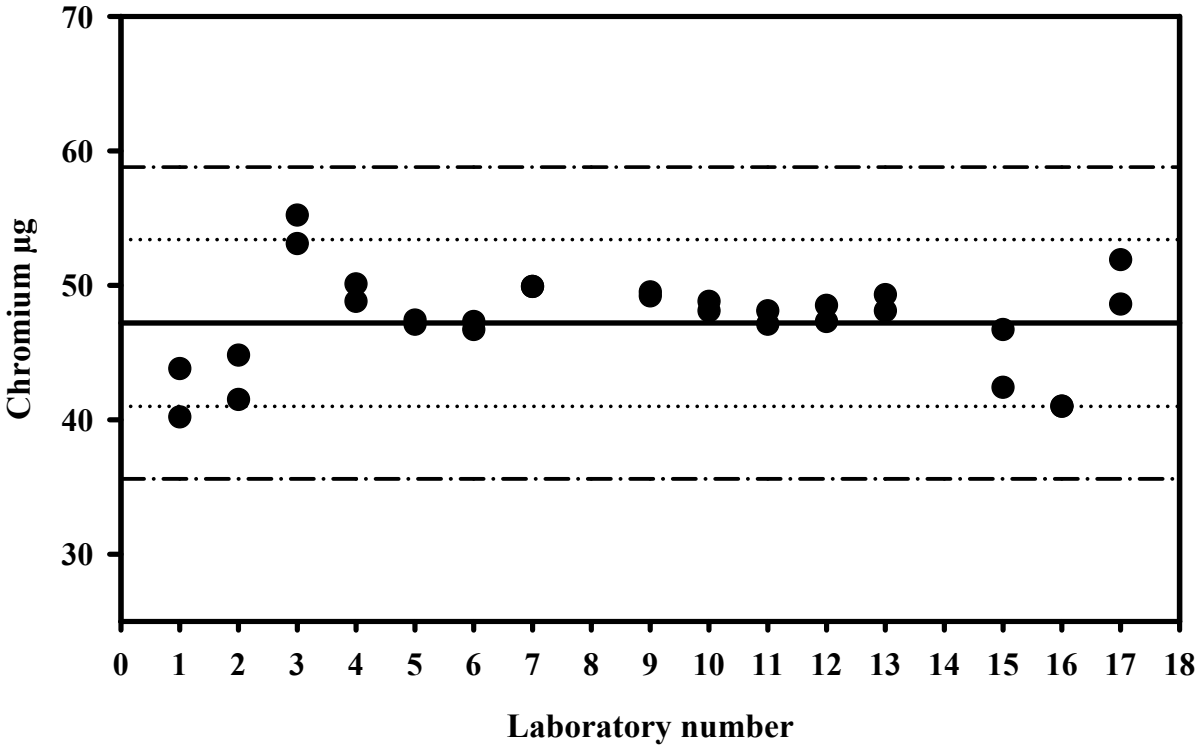
Cadmium - Series F

Reference value: 14.8 µg
Laboratory average: 14.5 µg



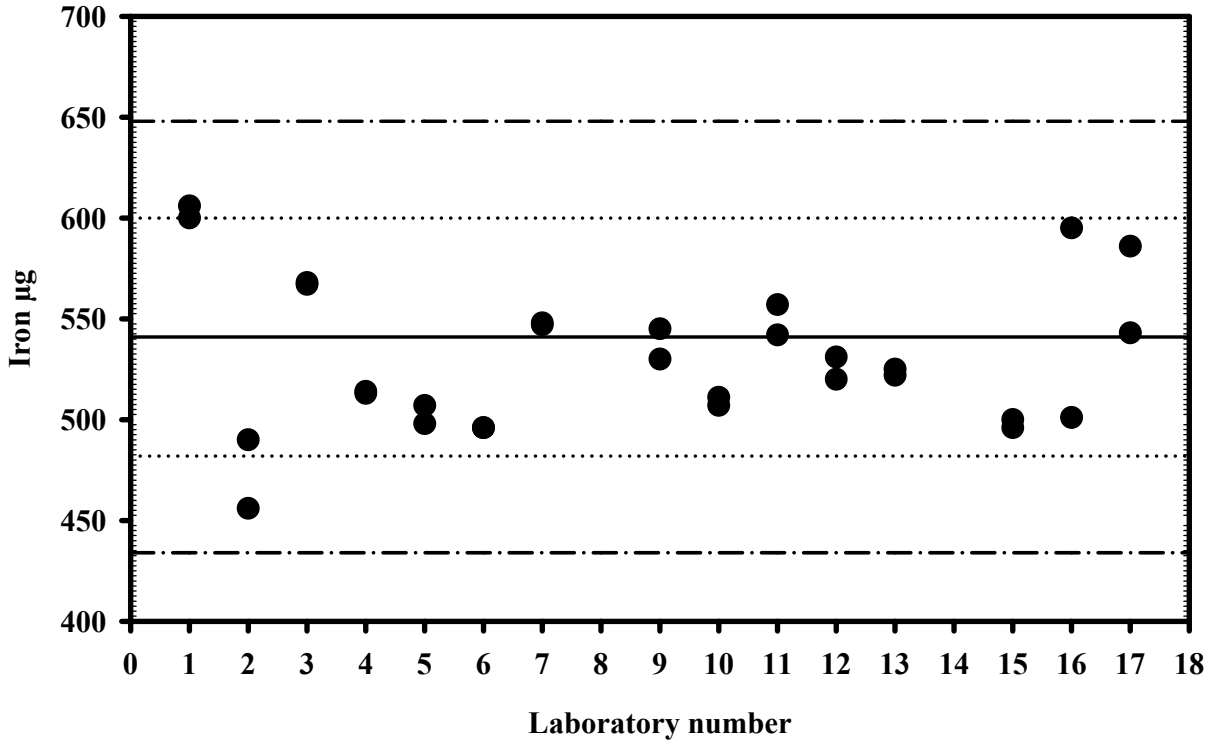
Chromium - Series F

Reference value: 47.2 µg
Laboratory average: 47.4 µg



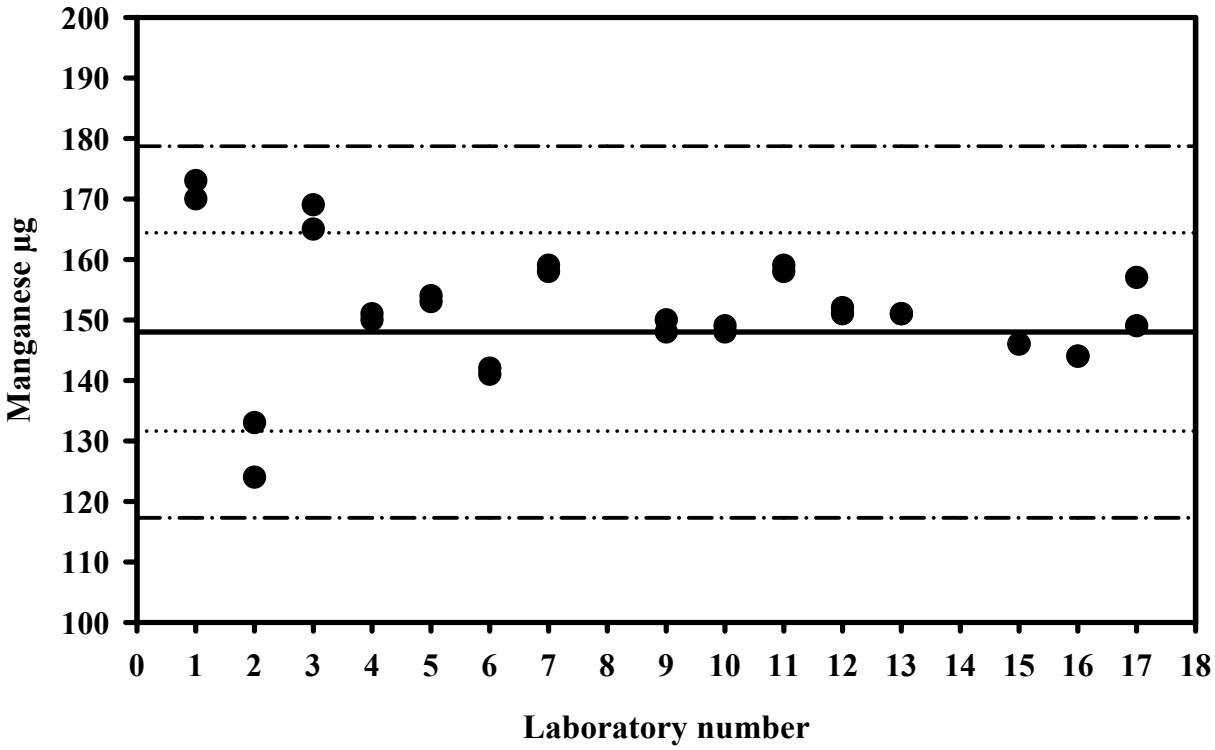
Iron - Series F

Reference value: 541 µg
Laboratory average: 527 µg



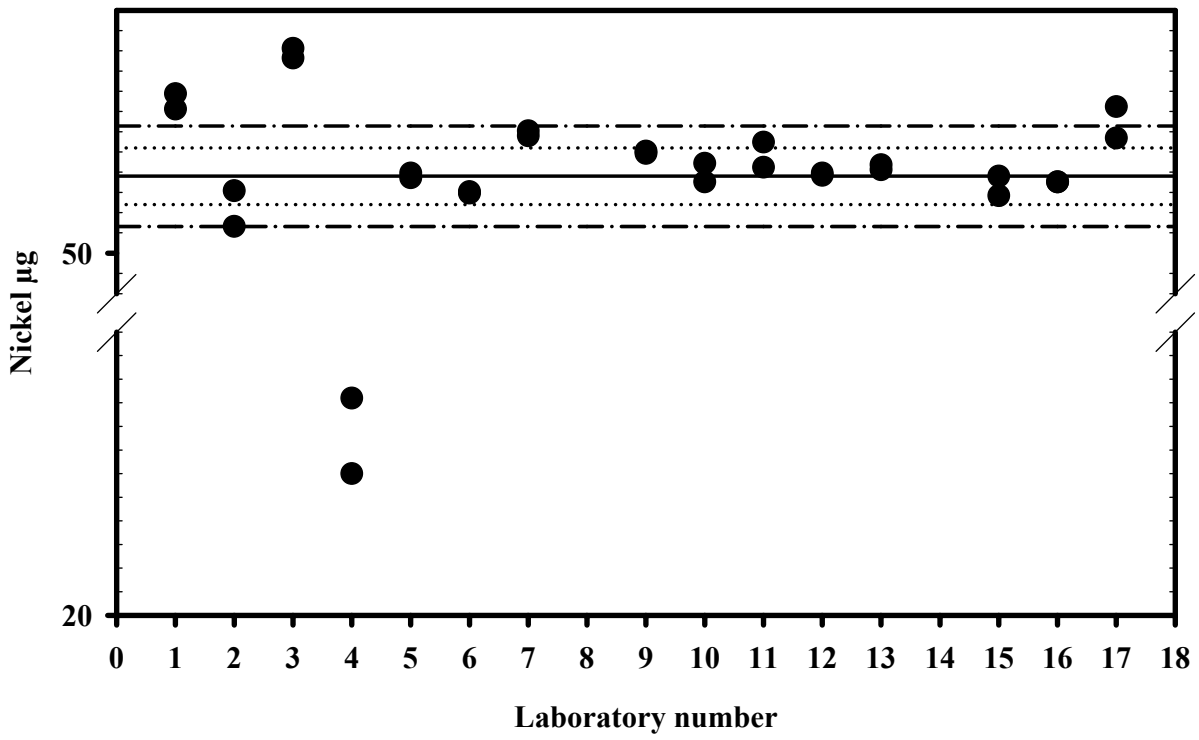
Manganese - Series F

Reference value: 148 µg
Laboratory average: 151 µg



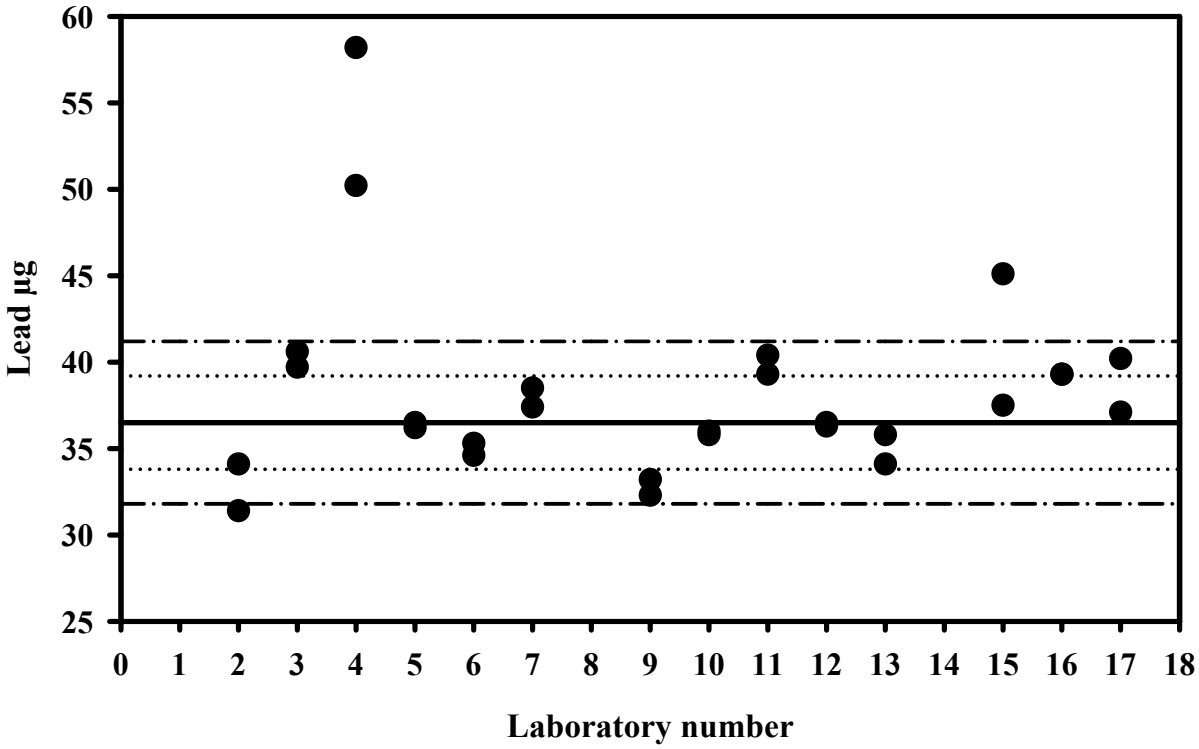
Nickel - Series F

Reference value: 59.5µg
Laboratory average: 62.1 µg



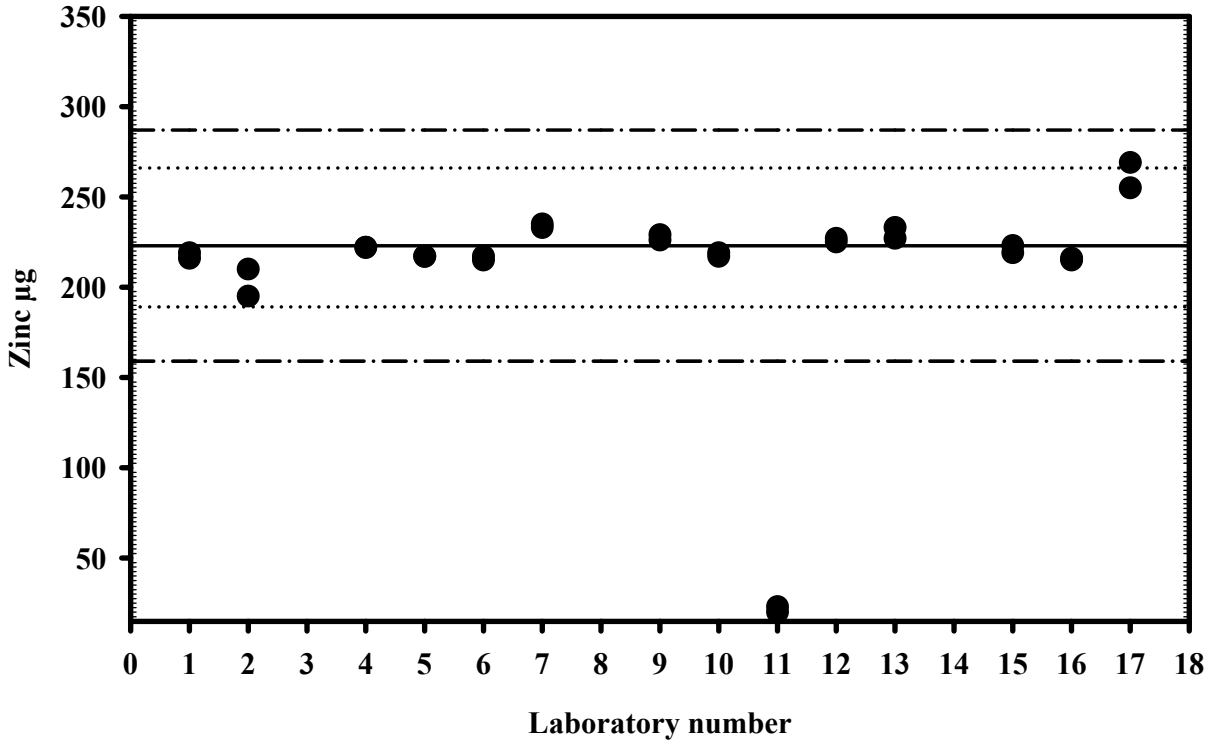
Lead - Series F

Reference value: 36.5 µg
Laboratory average: 36.2 µg

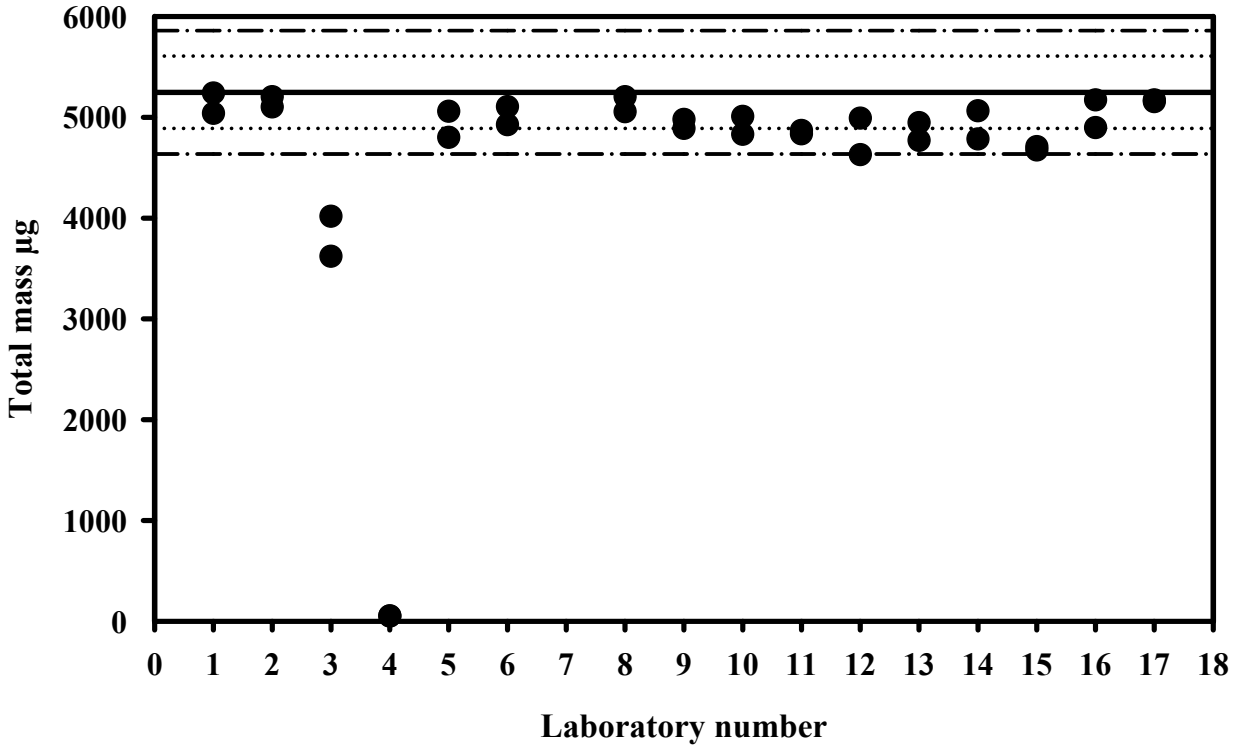


Zinc - Series F

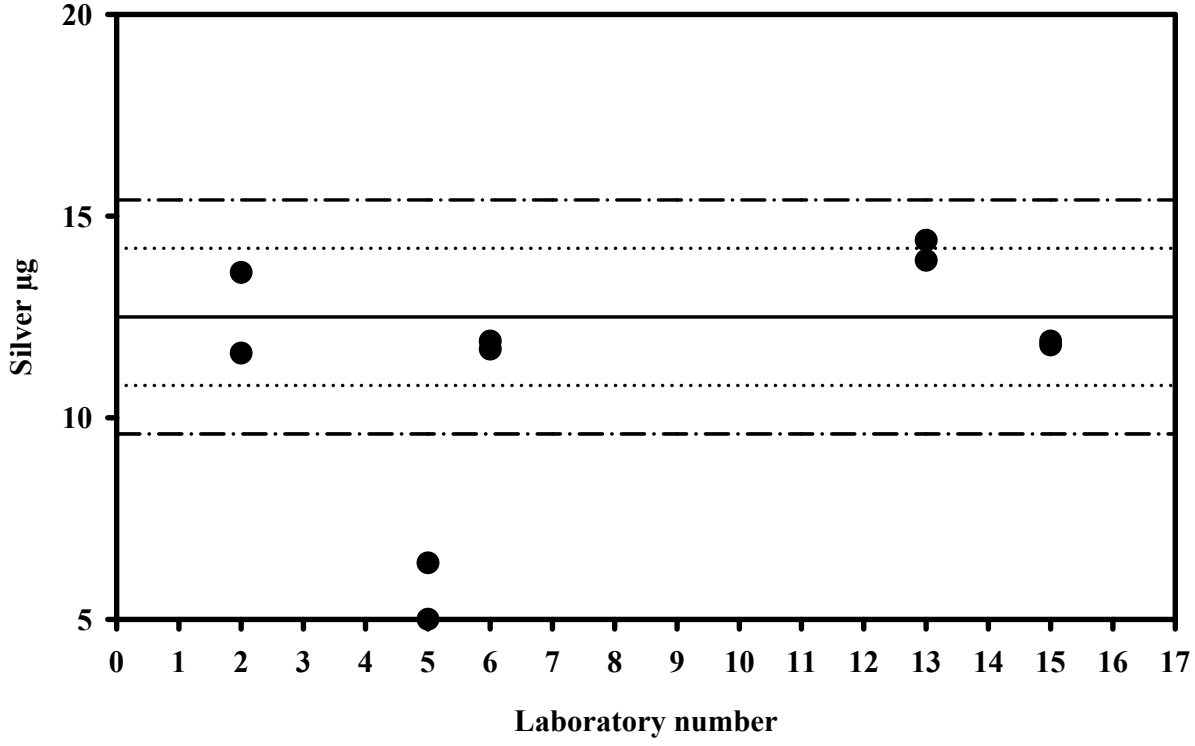
Reference value: 223 µg
Laboratory average: 224 µg



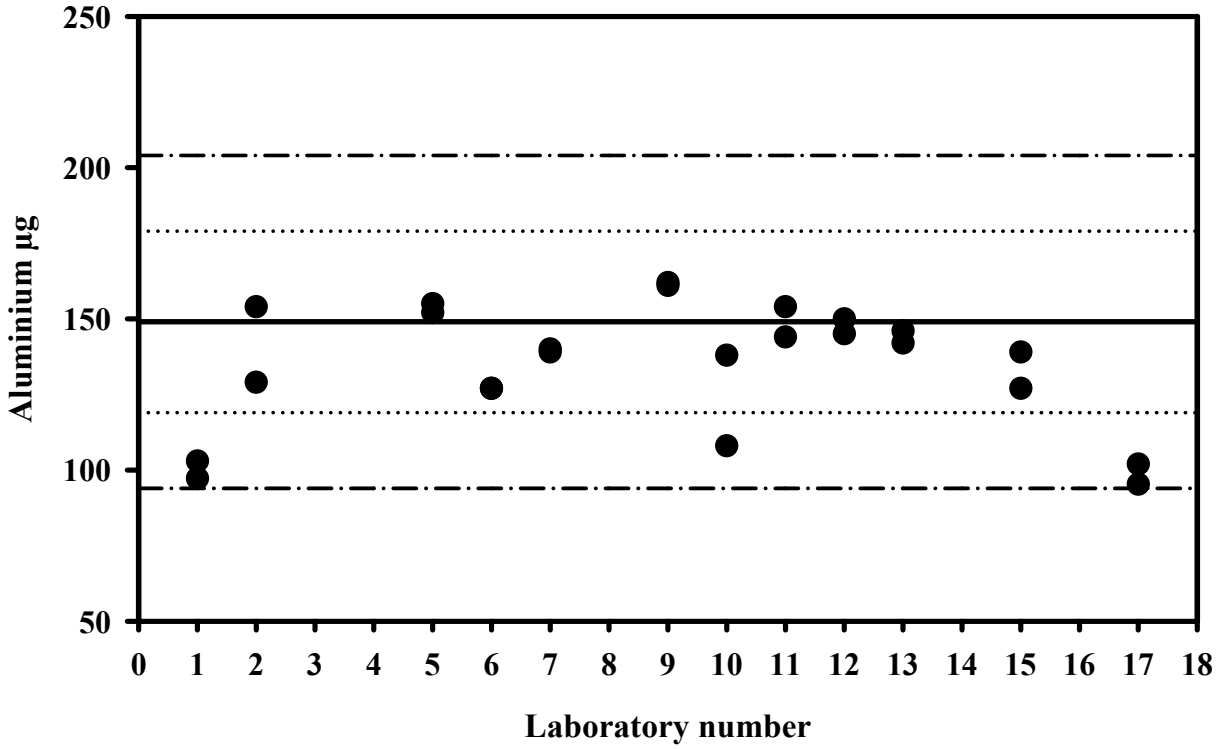
Total mass - Series Y
Reference value: 5247 µg
Laboratory average: 4934 µg



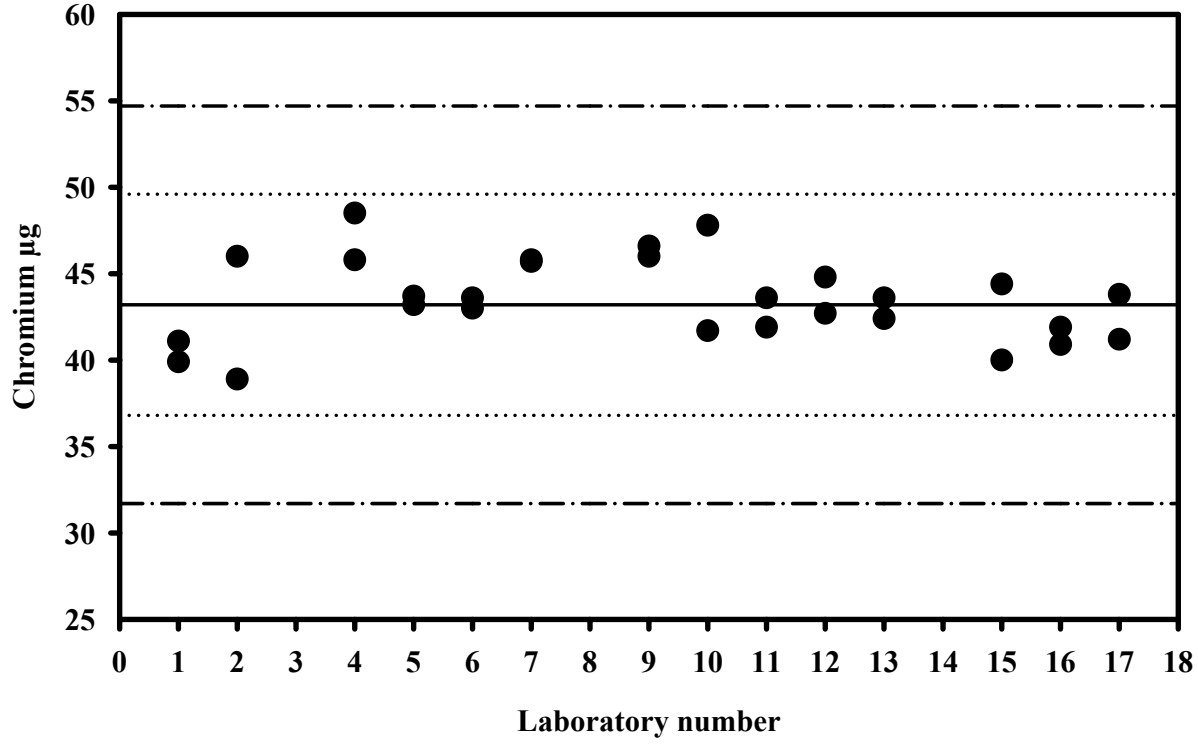
Silver - Series Y
Reference value: 12.5 µg
Laboratory average: 11.2 µg



Aluminium - Series Y
Reference value: 149 µg
Laboratory average: 138 µg



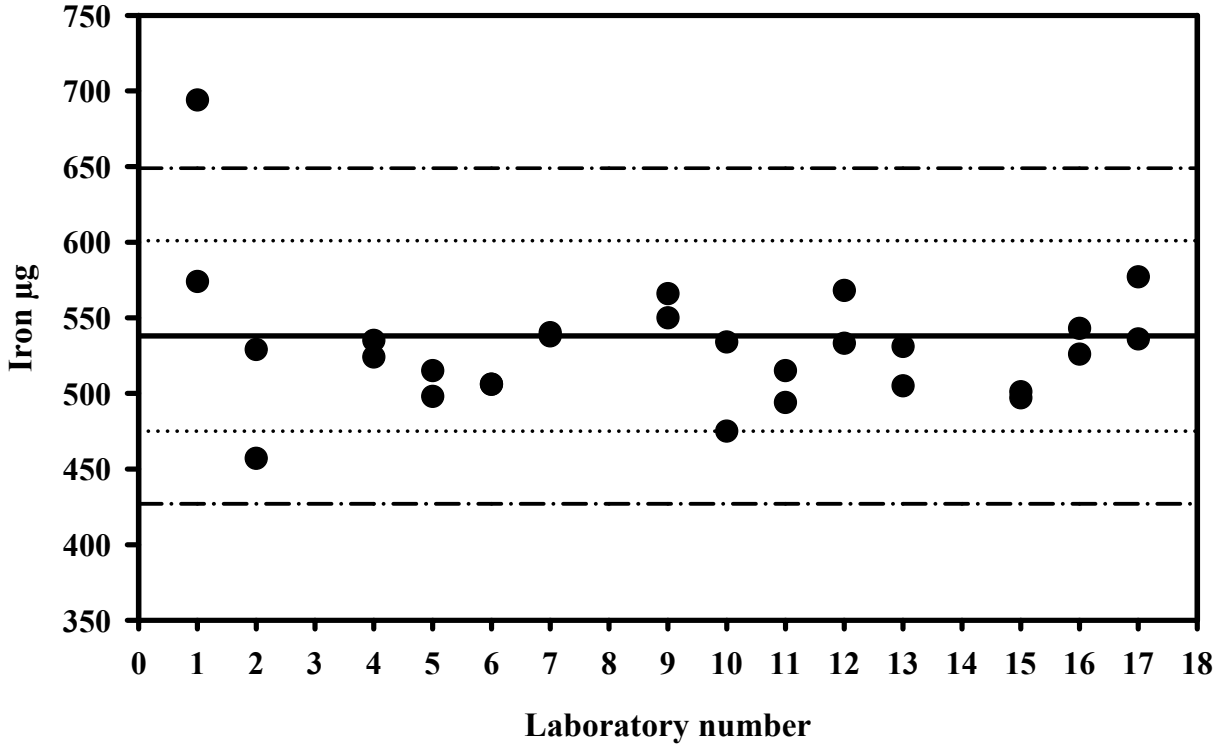
Chromium - Series Y
Reference value: 43.2 µg
Laboratory average: 43.5 µg



Iron - Series Y

Reference value: 539 µg

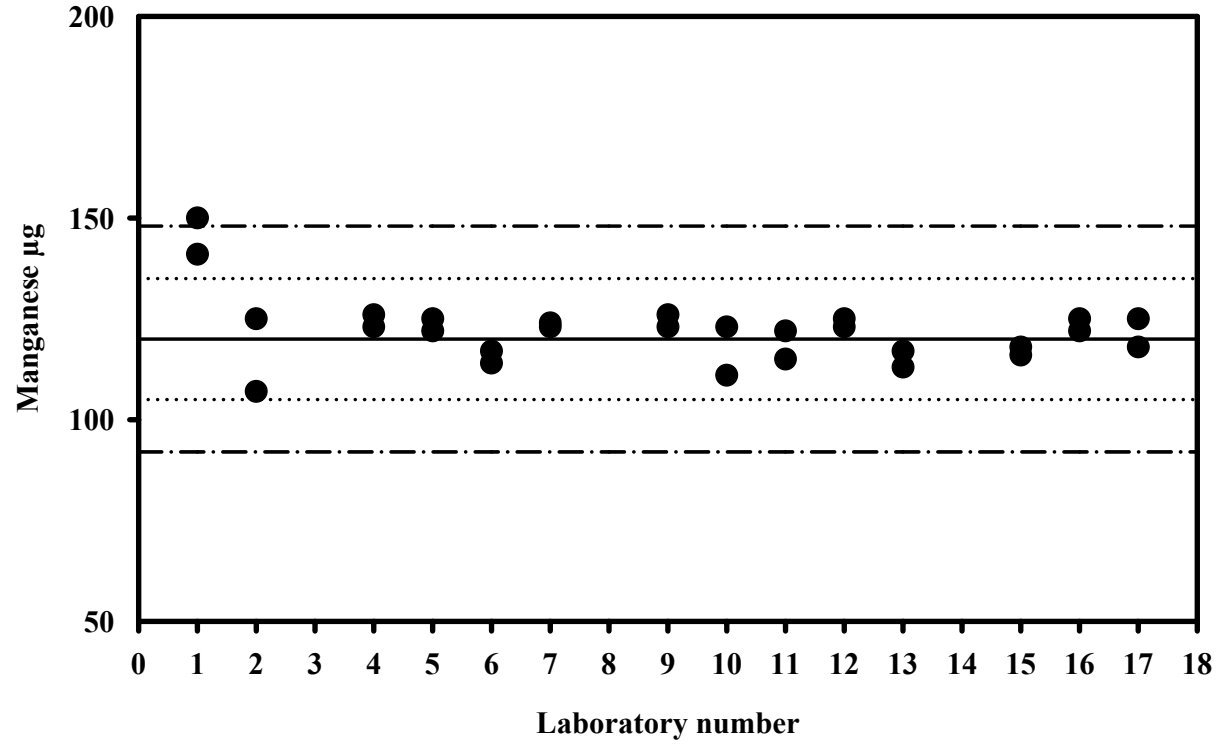
Laboratory average: 525 µg



Manganese - Series Y

Reference value: 120 µg

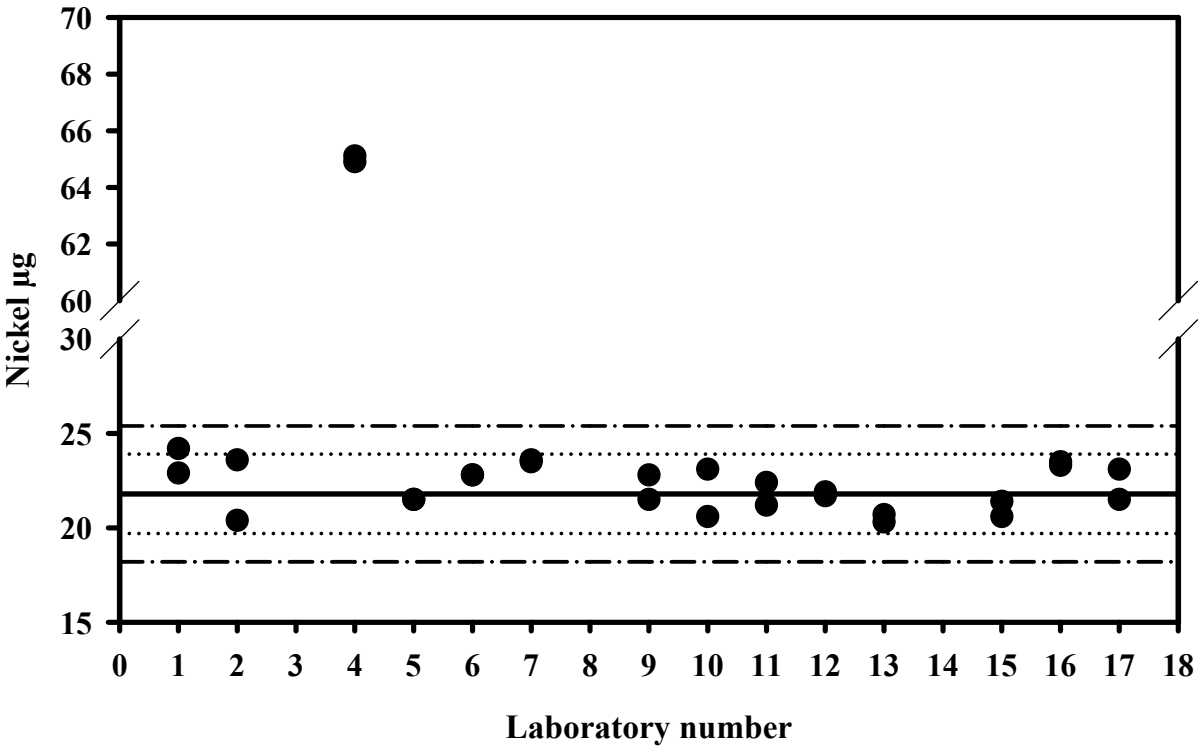
Laboratory average: 121 µg



Nickel - Series Y

Reference value: 21.8 µg

Laboratory average: 22.2 µg



Zinc - Series Y

Reference value: 21.1 µg

Laboratory average: 21.2 µg