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PERFORMANCE EVALUATION OF THE DEVICE FOR PM COLLECTION SMART SAMPLER

TECHNICAL REPORT

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INDEX

INTRODUCTION	pag. 5
OUTLINE OF THE STUDY	pag. 6
RESULTS	
Mass concentration	pag. 7
Inorganic anions and cations	pag. 9
Polycyclic aromatic hydrocarbons	pag. 15
Levoglucosane	pag. 20
Elements	pag. 22
CONCLUSIONS	pag. 26

INTRODUCTION

A study, commissioned by FAI Instruments, has been conducted by the Institute of Atmospheric Pollution of the National Research Council of Italy to evaluate the performances of the SMART SAMPLER. The SMART SAMPLER is a small device developed to collect atmospheric particles at a very low flow rate (0.5 L min⁻¹), typically operating over long sampling periods (1-2 months). The sampling is carried out on membrane filters, 37 mm in diameter, so as to allow the chemical analysis of the collected particles. Thanks to its features, the SMART SAMPLER can be used in two types of air quality studies: - evaluation of the long-term trend of the concentration of atmospheric particulate matter (PM) and of its chemical components; - determination of the spatial variability of the concentration of PM and its components, aimed to draw up concentration maps (useful, among others, to determine the strength of pollution sources).

This study is aimed to evaluate:

- 1. the repeatability of the measurements of PM and of some of its chemical components when the sampling is carried out by the SMART SAMPLER;
- for the same species, the difference in the concentration calculated when sampling with the SMART SAMPLER and when using instruments operating at the flow rate of 2.3 m³ h⁻¹, certified according to the European legislation;
- the existence and extent of possible artefacts when the sampling is carried out by the SMART SAMPLER.

The latter two objectives are justified by the following rationale. Every device and method for the sampling of atmospheric dust should not, in theory, alter the physico-chemical features of the sample in any way; it should also maintain unchanged the distribution of pollutants between the gaseous and the particulate phase. In real conditions, however, some inorganic species (mainly ammonium chloride and ammonium nitrate) and many organic species that can be grouped under the definition of "semi-volatile organic compounds" (chlorinated pesticides, polycyclic aromatic compounds and many other medium-to-high molecular weight species) undergo chemical equilibria between the two phases. These inter-conversion processes result in the following outcomes: overestimation of the mass concentration of particles due to adsorption of gaseous species on the collecting media and on the collected particles; underestimation of the mass concentration and variations in the chemical composition of PM due to the volatilization of inorganic salts or of organic species having sufficient vapour pressure (e.g.: release of nitric acid and ammonia from ammonium nitrate); variation in the chemical composition of PM due to displacement reactions (e.g.: reaction of nitric acid with sodium chloride particles, releasing hydrochloric acid and producing sodium nitrate). The overall effect of these artefacts cannot be predicted *a priori*, as it depends on the chemical composition of the sample and on many other parameters, including the temperature and relative humidity during the sampling period and the linear velocity of the air mass passing through the membrane filter. By convention, in the international scientific and legal community the reference for the determination of PM and its chemical components is the particulate amount collected on a filter, 37 or 47 mm in diameter, after a 24-h sampling carried out at the flow rate of 2.3 m³/h (European Standard UNI EN 12341:2014).

It is well known, however, that all the instruments used to collect atmospheric particulate matter are generally responsible for a considerable underestimation of both inorganic volatile salts and semi-volatile organic compounds, and that the higher is the PM fraction constituted by thermo-instable species, the higher is the underestimation. The artefacts occurring during long-duration, low flow rate samplings, such as those performed by the SMART SAMPLER, are certainly different from those observed in the case of the reference samplers. In the case of the SMART SAMPLER the linear velocity through the filter (37 mm in diameter) is about fifty times lower than in the case of the reference samplers (47 mm in diameter). This may cause differences in the concentration of PM and of its components measured by the SMART SAMPLERs and by the usual 2.3 m³/h samplers, differences that can be relevant, depending on the chemical species and the environmental conditions during the sampling.

OUTLINE OF THE STUDY

The study has been carried out at the facilities of the IIA-CNR (Research Area RM1, Montelibretti, Roma) during one year (August 2016 – July 2017). During the whole period PM samplings have been carried out by nine SMART SAMPLERs (long period) and by a reference sampler SWAM Dual Channel (24 hours). In order to be sure to collect enough PM amount, we decided to run the SMART SAMPLER for six weeks during the period August to October and March to July, and for four weeks during the period November to March, when the concentration in the study area generally increase. The choice of these time durations was based on the average concentrations detected at the site during the last ten years.

The nine SMART SAMPLERs placed in the field were equipped as follows:

- 3 with Teflon filters, aimed to determine the mass concentration of PM by the gravimetric procedure;
- 3 with quartz filters, aimed to determine anions and cation by ion chromatography (IC), elements by inductively-coupled plasma mass spectrometry (ICP-MS) and

levoglucosane by high-performance anion exchange chromatography with pulsed amperometric detection (HPAEC-PAD);

- 3 with quartz filters, aimed to determine polycyclic aromatic hydrocarbons (PAH) by high-resolution gas chromatography coupled with mass spectrometry (HRGC-MS).

The reference sampler has been equipped with quartz filters. The daily concentration of PM has been determined by the beta attenuation method.

To reduce the analytical costs, the 365 daily samples have been grouped as described below. By following this procedure, the number of analyses has been reduced, the representativeness of the daily samples has been maintained, and, for each analysis, the amount of particulate matter was comparable to the amount analysed for the SMART SAMPLER. The daily reference samples have been treated as follows:

- every filter has been cut into four sections (A,B,C,D);
- seven sections A (1/8 of each filter), corresponding to a sampling duration of one week, have been extracted all together and analysed for their ionic content by IC; the same procedure has been applied to sections B (1/8 of each filter), addressed to the analysis of the levoglucosane content by HPAEC-PAD, and to sections C (1/8 of each filter), addressed to the analysis of the elemental content by ICP-MS (regulated elements As, Cd, Ni, Pb and some other elements);
- seven sections D (half filter), corresponding to a sampling duration of one week, have been extracted all together and analysed for their content in polycyclic aromatic hydrocarbons by HRGC-MS.

RESULTS

Mass concentration

The mass concentration of PM_{10} during the nine measurement periods is shown in Figure 1 (results from the 3 co-located SMART SAMPLERs). The repeatability of the mass concentration measurements, expressed as standard deviation of the results obtained by the three co-located samples, was in the range 0.3 - 5.2 µg/m³, which corresponds to 2 - 24% of the collected mass amount.

These variability values are higher than those obtained for some PM components (e.g.: ions, see next paragraph) and are to be attributed to the low amount of dust collected on the filters (between 300 and 850 mg) and to the consequent variability in the results of the gravimetric determination.



Figure 1: Repeatability of the PM₁₀ mass concentration measurements (SMART SAMPLER)



Figure 2: PM_{10} mass concentration: SMART SAMPLER vs. SWAM dual channel.

The comparison between the SMART SAMPLER (average of the results obtained by the three co-located samplers) and the reference sampler (average of the daily concentration values during the considered period) is shown in Figure 2 (determination of the mass concentration of PM₁₀). The difference between the SMART SAMPLER data and the reference sampler data ranges between -10% and -26%. The yearly average concentration (from August 2016 to July 2017) was 20.8 μ g/m³ according to the SMART SAMPLERs and 25.8 μ g/m³ according to the reference sampler (20% difference). When evaluating these results, one must take into account that the SMART SAMPLER data are the average of three measurements, while the reference sampler data are the average of 45 daily values (30 values, in the case of the winter period). Moreover, it is worth underlining that the mass concentration is affected by all the possible sampling artefacts concerning the individual chemical species contributing to the PM mass. As already pointed out, these artefacts may be very different depending on the linear velocity through the filter and the time duration of the sampling.

Inorganic anions and cations

We considered the following chemical species: chloride, nitrate, sulphate, sodium, ammonium, potassium, magnesium, calcium. Their concentration during the nine measurement periods is shown in Figure 3 (results from the 3 co-located SMART SAMPLERs).

The repeatability of the measurements, expressed as standard deviation of the results obtained by the three co-located samples, was very good in all cases: the mean values referring to the whole study are below 4% for nitrate, sulphate and ammonium and between 4% and 7.5% for the other species. These results indicate that the samplings carried out by the SMART SAMPLERs are very repeatable. It follows that the much less satisfactory results obtained when measuring the mass concentration of PM_{10} are to be attributed to a lower precision of the measurement technique (gravimetric procedure in the case of the mass amount, ion chromatography in the case of ions).

The comparison between the SMART SAMPLER (average of the results obtained by the three co-located samplers) and the reference sampler (average of the daily concentration values during the considered period) for the measurement of the ionic species is shown in Figure 4.

To make an accurate assessment of the SMART SAMPLER performances we should consider both the thermal stability and the size distribution of each species. Concerning the thermal stability, ammonium chloride and ammonium nitrate, among the inorganic salts, are affected by the thermodynamic conditions during the sampling (temperature, relative humidity) and may be released from the filter. To evaluate the size distribution we can consider the data reported in Figure 5, which refer to the size distribution of ions obtained from a sampling carried out during April 2017, at the same site of this study, by using a multi-stage impactor.



Figure 3: Repeatability of the concentration measurements of inorganic ions (SMART SAMPLER).



Figure 4: Concentration of inorganic ions: SMART SAMPLER vs. SWAM dual channel.



Figure 5: Size distribution of inorganic ions.

We can notice that some species (ammonium, sulphate; orange bars in Figure 5) are distributed almost exclusively in the fine fraction of PM (below 2.5 μ m). Ammonium sulphate is, in fact, a secondary species, produced by atmospheric reactions and thus found in the nucleation and accumulation mode only. Some other species (chloride, sodium and magnesium; blue bars in Figure 5) are mainly distributed in the coarse size fraction (above 2.5 μ m), in agreement with their main source, the sea-spray. Calcium (violet bars in Figure 5), mainly generated through erosion and soil dust ri-suspension, also shows a size distribution in the coarse fraction of PM, characterized by a relevant contribution in the fraction above 10 μ m. Potassium (green bars in Figure 5) shows a bimodal distribution that reflects its two main sources: biomass combustion for the fine fraction and soil re-suspension for the coarse fraction. The first source is on during the cold season, while the second one is generally stronger during the warm season, because of soil dryness.

The size distribution of nitrate also shows a seasonal variability: during the spring, summer and autumn this species can be found mainly in the coarse fraction (re-suspension of soil dust; reaction between nitric acid and sodium chloride leading to the formation of sodium nitrate), during winter periods of atmospheric stability, instead, the amount in the fine fraction, due to the formation of ammonium nitrate, prevails (also this second contribute is visible, to a minimal extent, in Figure 5).

The data in Figure 4 and the comparison of the yearly average concentrations, reported in Figure 6, show that a very good agreement between SMART SAMPLER and reference sampler is obtained for sulphate, a thermostable species contained almost exclusively in the fine fraction of PM_{10} , and for potassium, a thermostable species contained mainly in the fine fraction of PM_{10} , especially during the warm months. The ratio \mathbf{R} between the yearly average concentrations yielded by the two systems (SS/REF) is, in facts, as high as 1.02 for sulphate and 1.00 for potassium.

For sodium, magnesium and, most of all, calcium, the concentrations obtained by using the SMART SAMPLER are always higher than the concentrations yielded when using the reference sampler, with \mathbf{R} values of 1.23 (sodium), 1.27 (magnesium) and 1.54 (calcium). This is probably due to a different performance of the PM₁₀ impactors. These parts of the sampling heads retain particles have aerodynamic diameter higher than 10 µm, while let all particles smaller than this size pass through and be collected on the filter. It is plausible that the impactors of the SMART SAMPLER, which operates at a flow rate about 80 times lower than the reference sampler, have a different and less sharp cut-off curve (the curve describing the impact probability on the impactor plate as a function of the aerodynamic diameter of the particles). The SMART SAMPLER impactors could therefore allow the collection of a higher amount of particles greater than 10 µm, with respect to the impactor of the reference samplers.

In the case of nitrate and ammonium, the number of tests carried out during this study is not sufficient to obtain a reliable interpretation of the small differences between SMART SAMPLERs and reference samplers (\overline{R} = 0.91 and \overline{R} = 0.87, respectively). Ammonium ion is chemically bound both to sulphate, to form a thermostable species, and to nitrate and chloride, which, instead, may release their gaseous precursors. In the geographical area of the study, nitrate ion is mainly bound to sodium, to form a thermostable species. However, during the winter period, the contribution of ammonium nitrate may become relevant. The stability of ammonium nitrate during the sampling and the release of nitric acid and ammonia still have to be completely understood; these processes are certainly influenced by the temperature and relative humidity during the sampling (which determine the deliquescence of the salts), the linear velocity of the air mass passing through the filter and the duration of the sampling. All these parameters are generally different for the two samplers. Moreover, for nitrate in the coarse fraction we have the same situation related to the cutsize of the impactor that we have described for sodium, magnesium and calcium.

Atmospheric chloride can be found in the form of sodium chloride, ascribable to sea-spray, and in the form of ammonium chloride, a secondary species that undergoes the same instability process described for ammonium nitrate. For this species the value of \bar{R} was 0.58.



Figure 6: One-year (August to July) average concentration of the ionic species: SMART SAMPLER vs. SWAMdc.

Polycyclic aromatic hydrocarbons (PAH)

Ten PAH congeners, having 4, 5 and 6 condensed rings, have been considered in this study: fluorantene, pirene, benzo(a)antracene, crisene (4 rings), benzo(b)fluorantene, benzo(b+j+k)fluorantene, benzo(e)pirene, benzo(a)pirene, dibenzo(a,h)antracene (5 rings), indeno(1,2,3,c,d)pirene, benzo(g,h,i)perilene (6 rings).

Among these, benzo(a)pirene is particularly important as it has been classified as human carcinogen (class 1) by the International Agency for Research on Cancer (IARC). The European legislation (European Directive 2008/50/EC) has set a target value of 1 ng/m³ for this species (yearly average).

The concentration of PAHs during the nine measurement periods is shown in Figure 7 (results from the 3 co-located SMART SAMPLERs). It is apparent that all the congeners show a clear seasonal trend, characterized by much higher concentration during the cold period. This can be due to the possible decomposition of PAHs by ozone, whose concentration increases during the summer, as well as to the contribution of a winter source due to biomass combustion in domestic heating.

The repeatability of the measurements for the ten individual PAH congeners, expressed as standard deviation of the results obtained by the three co-located samples, was in the range 12% - 22% (average data referring to the whole study period). The standard deviation for the sum of the ten congeners (Figure 8) was 10%. Considering the very low concentrations and the complexity of the analytical procedure, these results are to be considered as very good and confirm those obtained for the ionic species.

The comparison between the SMART SAMPLER (average of the results obtained by the three co-located samplers) and the reference sampler (average of the daily concentration values during the considered period) for the measurement of PAHs is shown in Figure 9.

In order to correctly evaluate the results obtained for PAHs, it is necessary to consider, as already underlined, that these species are unstable because of their easy oxidation by atmospheric oxidants, among which ozone. Moreover, some of these compounds, mainly the lower molecular weight congeners, are semi-volatile. In air quality studies, some solutions have been identified to obtain the best correspondence between PAH concentration in the atmosphere and in the collected sample: ozone scrubbers, placed inside the inlet, are used to reduce sample oxidation; back-up polyurethane foams (PUF) are used to recover the amount released during the sampling; finally, the length of the sampling phase is kept as low as possible. The SMART SAMPLERs differ from the reference samplers because of both higher duration of the sampling periods and lower flow rate. A long sampling duration, in theory, could favour the release of the most volatile congeners, while, conversely, a lower flow rate could favour their stability.





Figure 7: Repeatability of the concentration measurements of PAHs (SMART SAMPLER).



Figure 8: Repeatability of the concentration measurements of PAHs: sum of the 10 congeners (SMART SAMPLER).

The data reported in Figure 9 show that we obtained different results for the different PAH congeners. For the lighter, 4-ring species (fluorantene, pirene, benzo(a)antracene, crisene), the concentration determined when sampling with the SMART SAMPLERs was higher than in the case of the reference sampler. For the other 5-ring and 6-ring congeners, instead, the reverse situation occurred. Higher concentration when sampling with the reference instrument were obtained also when considering the sum of the 10 congeners (Figure 10), given the higher contribution of the more complex congeners to the total concentration.









2,0

1,8

1,6 1,4

1,2 ng/m³

1,0

0,8

0,6 0,4

0.2

0.0







Figure 9: Concentration of PAHs: SMART SAMPLER vs. SWAM dual channel.



Figure 10: Concentration of PAHs (sum of the 10 congeners): SMART SAMPLER vs. SWAM dual channel.

The ratio \mathbf{R} (SS/REF) between the 1-year average concentration yielded by the two systems, compared in Figure 11, is above one for the 4-ring species (1.69, 1.70, 1.04 and 1.20 for fluorantene, pirene, benzo(a)antracene and crisene, respectively), while it is lower for the 5-ring and 6-ring species (0.79, 0.55, 0.43, 0.53, 0.74 and 0.60 for benzo(b+j+k)fluorantene, benzo(e)pirene, benzo(a)pirene, indeno(1,2,3,c,d)pirene, dibenzo(a,h) antracene and benzo(g,h,i)perilene, respectively). From these results, it seems that when the sampling is carried out by using the SMART SAMPLER a decrease of the stripping effect, due to the lower linear velocity of the air masses through the filter, prevails for the more volatile PAH congeners, while a prevalence of the decomposition due to the length of the sampling duration is observed for the heavier species.



Figure 11: One-year (August to July) average concentration of the PAHs: SMART SAMPLER vs. SWAM dual channel.

Levoglucosane

The concentration of levoglucosane during the nine measurement periods is shown in Figure 12 (results from the 3 co-located SMART SAMPLERs). As already highlighted in the case of PAHs, the concentration of this compound shows a clear seasonal pattern: during the winter the concentration is about one order of magnitude higher than during the warm period. Levoglucosane is, in fact, a tracer of biomasses, that are widely used for domestic heating during the cold season and, sporadically, for cooking food (barbecue) during the summer.

The repeatability of the measurements of levoglucosane, expressed as standard deviation of the results obtained by the three co-located samples, was very satisfactory: the values were in the range 0.5% - 10%, with an average of 5.3% (whole study period).

The comparison between the SMART SAMPLER (average of the results obtained by the three co-located samplers) and the reference sampler (average of the daily concentration values during the considered period) for the measurement of levoglucosane is shown in Figure 13. This species is a stable compound and, being a combustion product, its size distribution is mainly in the fine fraction of atmospheric dust. For these reasons, levoglucosane do not suffer either from the different duration of the samplings, or from the different cut-size of the impactors. The results of the comparison are in fact very good, and the value of $\mathbf{\bar{R}}$ (SS/REF) is 1.03.



Figure 12: Repeatability of the measurements of levoglucosane (SMART SAMPLER).



Figure 13: Levoglucosane concentration: SMART SAMPLER vs. SWAM dual channel.

Elements

In the case of the elemental concentration, the evaluation of the repeatability has been carried out on 4 groups of samples only. For the other samples, due to the very low concentration levels in the study area, we preferred to group the three replicate filters into one sample, to be compared with the reference sample. The results obtained for the three co-located samplers are shown in Figure 14 for the four regulate elements (arsenic, cadmium, nickel, lead) and in Figure 15 for other 6 elements that can be found in the same atmospheric concentration range (copper, rubidium, manganese, molybdenum,, vanadium, caesium).

The repeatability of the measurements was very good; for the ten elements, the standard deviation of the results obtained by the three co-located samples was in the range 6% - 15% (average value during the four considered periods). In particular, the repeatability of the measurements of the four regulated elements was 9% for Pb, 10% for As e Cd, 11% for Ni. We had excellent results for manganese (average repeatability: 6%); for caesium, whose concentration level is of the order of a few tenths of nanogram per cubic meter, the average repeatability (14%) was also very satisfactory.



Figure 14: Repeatability of the measurements of regulated elements (SMART SAMPLER).



Figure 15: Repeatability of the measurements of some elements (SMART SAMPLER).

The comparison between the SMART SAMPLER (average of the results obtained by the three co-located samplers) and the reference sampler (average of the daily concentration values during the considered period) for the measurement of elements is shown in Figure 16 for the regulate elements (arsenic, cadmium, nickel, lead) and in Figure 17 for the other elements (copper, rubidium, manganese, molybdenum,, vanadium, caesium). In all cases, the concentration in the samples obtained by the SMART SAMPLERs and the reference sampler were in good agreement.

In particular, for the elements that are mainly in the fine fraction of PM (Cd and Pb, among the regulated species, Rb e V, among the others) we obtained very good results: the ratio \bar{R} (SS/REF) was 1.01 for Cd, 1.09 for Pb, 1.06 for Rb and V.

For the other elements, characterized by a relevant amount in the coarse size fraction, the concentration detected in the samples collected by the SMART SAMPLERs were slightly higher than those detected in the samples collected by the reference sampler, with \mathbf{R} values in the range 1.10 - 1.32. As discussed for the ionic species, these difference are probably related to a different performance of the impactors. It is worth noting that these very low differences do not impair the use of the SMART SAMPLER in homogeneous measurement networks, where the main required feature is the repeatability of the samplings.



Figure 16: Concentration of regulated elements: SMART SAMPLER vs. SWAM dual channel.



Figure 17: Element concentrations: SMART SAMPLER vs. SWAM dual channel.

CONCLUSIONS

The results of this study, aimed to evaluate the performance of the SMART SAMPLERs, have led to the following conclusions:

- the repeatability of the concentration measurements (3 co-located instruments, average of the 9 measurements carried out for each chemical species or groups of species) was about 20% for PM₁₀ concentration, 4-8% for the inorganic ions, 12-22% for PAHs, 5% for levoglucosane and 6-15% for the elements; the lowest values (4-5%) are a good estimate of the true repeatability of the sampling phase, while the other values are most likely due instability/decomposition phenomena for the considered compounds and/or to the analytical uncertainty due to the low amounts of the collected dust;
- the comparison with the SWAM Dual Channel automatic sampler, considered as reference, was very satisfactory for the chemical components that are stable and mainly in the fine fraction of PM₁₀ (sulphate, potassium, levoglucosane, cadmium, lead, rubidium, vanadium); for the species that are mainly in the coarse faction, the concentration are generally higher when the samplings are carried out by using the SMART SAMPLERs, probably because of a different slope of the cut-off curve of the impactors; for the species that undergo desorption/decomposition/release processes, the performance of the SMART SAMPLERs differ from species to species and can vary according to the sampling conditions; the mass concentration of PM₁₀ determined when using the SMART SAMPLER was lower (on average, about 20%) than the value obtained when using the reference sampler;
- as the mass concentration of PM₁₀ was lower when using the SMART SAMPLERs while the concentration of ions was generally in agreement, it can be assumed that some release phenomena occur for macro-components not considered in this study. These macrocomponents (i.e. species that constitute more than 1% of the total mass) generally include some elements released from the soil (silicon, aluminium), elemental carbon and organic species (taken together). For crustal elements, stable and almost totally in the coarse fraction of PM₁₀, and for elemental carbon, also very stable, this behaviour cannot be expected. We therefore believe that the organic fraction of particulate matter may undergo relevant release phenomena; this fraction is quantitatively considerable (up to 50%) and can be thus responsible for the decrease in PM mass concentration detected when sampling with the SMART SAMPLER.

The SMART SAMPLERS have been developed to evaluate the spatial distribution of atmospheric particulate matter and of its chemical components. For this reason, their ability to carry

out repeatable samplings is the most desirable features, as it guarantees the reliability of the concentration maps. It is worth noting that the observed disagreement with the reference sampler, due to the low sampling flow rate and the different cut-size of the impactor, become irrelevant when the sampling network is comprised of identical SMART SAMPLER devices.

Dott.ssa Cinzia Perrino

subolours

Montelibretti, 15 november 2017

APPENDIX

Translation of some terms used in the Figures

ITALIAN	ENGLISH
Ago	August
Set	September
Ott	October
Nov	November
Dic	December
Gen	January
Feb	February
Mar	March
Apr	April
Mag	May
Giu	June
Lug	July
CLORURO	CHLORIDE
NITRATO	NITRATE
SOLFATO	SULPHATE
SODIO	SODIUM
AMMONIO	AMMONIUM
POTASSIO	POTASSIUM
MAGNESIO	MAGNESIUM
CALCIO	CALCIUM
LEVOGLUCOSANO	LEVOGLUCOSANE
ARSENICO	ASSENIC
CADMIO	CADMIUM
NICHEL	NICKEL
PIOMBO	LEAD
RAME	COPPER
RUBIDIO	RUBIDIUM
MANGANESE	MANGANESE
MOLIBDENO	MOLIBDENUM
VANADIO	VANADIUM
CESIO	CAESIUM