

## Analysis of tellurium by spark source mass spectrometry\*

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### Analyse von Tellurium mit Hilfe der Funkenionisations-Massenspektrometrie

**Summary.** Three tellurium standards were analyzed by both spark source mass spectrometry (SSMS) and graphite furnace atomic absorption spectrophotometry (GFAAS). From these results relative sensitivity coefficients (RSC) for spark source mass spectrometry were derived for 24 elements.

With these RSC's SSMS results within a factor 1.5 from the GFAAS values could be obtained for the determination of various impurities in tellurium. A comparison is made between SSMS, GFAAS and glow discharge mass spectrometry (GDMS) for the analysis of 4–6 N tellurium samples.

### Introduction

Materials such as Te, Cd, In, Ga, As and Se form the basis of modern electronics. Trace impurities in electronic materials can seriously affect the device yield and performance. The requirements for higher purity of these materials are constantly increasing, which emphasizes the need for survey analysis methods in order to check the chemical composition. Ideally, the analytical technique used to study these materials should have the following features: (1) panoramic elemental capability (non-metals like S, Cl, Br, I, P included), (2) good detection limits ( $\leq 10$  ppb), (3) uniform sensitivity and maximum selectivity, (4) short analysis time and (5) good precision and accuracy. At present, only 2 techniques are commercially available which satisfy the three first and for high purity materials most important requirements at the same time, namely spark source mass spectrometry (SSMS) and glow discharge mass spectrometry (GDMS). If the total number of elements in one analysis (ca. 70) is considered, then both methods are relatively fast in comparison with a technique such as for example atomic absorption spectroscopy (AAS). With both techniques results are obtained which are typically correct within a factor of 3 (standardless analysis). Better results can be obtained when relative sensitivity coefficients (RSC) are known.

\* Dedicated to Prof. Dr. G. Tölg on the occasion of his 60th birthday

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In this work, SSMS was used to analyze tellurium. In order to improve the accuracy RSC's were determined by analysis of standard samples. For Te no standard reference materials are commercially available. This was solved by self-preparing Te samples which satisfy the principal requirements of a standard, namely the impurities should be homogeneously distributed and be present at a fairly high concentration level (1–100 ppm), so as to allow the use of an accurate method such as GFAAS for standardization. Three Te standards were prepared in our laboratory. Using these standards for SSMS analysis, it was possible to determine RSC values. These RSC's can also be used for high purity (6 N) samples since the RSC is independent of the concentration over at least 5 orders of magnitude [1–4].

A 4 N Te sample was analyzed by SSMS, GFAAS and GDMS in order to study the potential of these techniques for the analysis of Te.

Also, the analysis of high purity Te (6 N) by SSMS is presented using the RSC correction. The SSMS results are compared with the corresponding GFAAS and GDMS results.

In the literature, few reports are available on the analysis of Te by SSMS [5–7] and no relative sensitivity coefficients are given in any of these reports.

### Experimental

Three laboratory standards of tellurium were prepared. For this purpose impure (4 N) and pure solid samples of different composition were pulverized and sieved until fine powders with a grain size of circa 100 mesh were obtained. These powders were further mixed in various proportions so as to obtain materials with suitable impurity concentrations of various elements. Each powder was homogenized by shaking it for several hours in a 3-dimensional shaker (Turbula). Most operations were carried out in an inert atmosphere. The composition of these "standards" was determined by GFAAS. The results are shown in Table 1. Each result is the mean of GFAAS measurements on 4 preparations of each sample. The precision of these measurements is also indicated in Table 1 ( $\pm 1$  standard deviation). For some elements rather imprecise results were obtained which are due to the heterogeneous distribution of these elements in the sample.

SSMS was carried out with a JEOL JMS-01-BM2 double focusing instrument with an automatic spark control system. The detection was performed on Ilford Q2 ion sensitive

**Table 1.** Composition (ppmw) of the Te standards determined by GFAAS

Element	Concentration (ppmw)		
	Standard 1	Standard 2	Standard 3
Na	1.04 ± 0.03	0.93 ± 0.04	—
Mg	0.07 ± 0.01	0.07 ± 0.02	<0.03
Al	0.375 ± 0.007	0.13 ± 0.03	—
Si	2.6 ± 0.9	<0.5	—
K	0.067 ± 0.015	0.09 ± 0.02	—
Ca	0.31 ± 0.02	0.08 ± 0.01	—
Cr	1.49 ± 0.06	1.30 ± 0.07	0.44 ± 0.03
Mn	0.17 ± 0.01	0.27 ± 0.02	—
Fe	15.2 ± 0.8	13.0 ± 0.2	4.4 ± 0.2
Co	0.053 ± 0.006	0.028 ± 0.003	—
Ni	1.6 ± 0.1	5.6 ± 0.2	0.35 ± 0.04
Cu	0.94 ± 0.06	—	0.34 ± 0.04
Zn	2.57 ± 0.07	0.41 ± 0.03	—
As	8.7 ± 0.5	—	—
Se	11.0 ± 0.3	—	—
Mo	0.69 ± 0.08	0.20 ± 0.02	—
Ru	—	0.11 ± 0.02	—
Rh	—	5.1 ± 0.2	—
Pd	—	2.6 ± 0.1	—
Ag	0.40 ± 0.02	36.0 ± 0.04	0.11 ± 0.01
Cd	0.057 ± 0.006	—	—
In	0.63 ± 0.06	0.22 ± 0.03	—
Sn	0.357 ± 0.006	0.39 ± 0.06	0.26 ± 0.05
Sb	2.5 ± 0.1	15.0 ± 0.4	0.71 ± 0.17
Ir	—	0.66 ± 0.05	—
Au	<0.03	21.0 ± 0.3	—
Pb	2.16 ± 0.01	2.6 ± 0.2	1.3 ± 0.1
Bi	<0.05	0.25 ± 0.03	<0.05

plates. The experimental conditions selected for the Te analysis are summarized in Table 2. On each photoplate 12 exposures were recorded in the range  $10^{-3}$  to 300 nC in steps increasing by a factor of 3. Only for the analysis of the high purity Te sample (6 N) a higher exposure of 600 nC was collected. After development, the photoplates were read by a single beam densitometer (JMD-2C, JEOL) coupled to a microcomputer system. Data reduction was performed on a DEC VAX 11/780 computer using a program package developed by Pilate et al. [8]. The transmission profiles were integrated using the Hull function [9]. Solid Te samples can be analyzed directly by SSMS after selecting pieces (electrodes) of suitable dimensions (3–10 mm length, 0.3–2 mm width, 0.3–2 mm thick). The Te powders are pressed (12 tons) into cylindrical electrodes of circa 1.5 mm diameter and 10 mm length. Before analysis, the electrodes were pre-sparked in order to remove surface contamination. Memory effects due to previous analyses of other matrices were reduced by using an ion source exclusively reserved to Te matrices.

A Perkin Elmer 5100 graphite furnace atomic absorption spectrophotometer (GFAAS) was used for accurate determinations of the impurity elements in Te. It is a fully automated instrument with an HGA-600 graphite furnace and an AS-60 autosampler. It consists of the spectrophotometer and of the Perkin-Elmer Series 7000 Professional Computer as the controller. It is also equipped with the Zeeman background correction system. For AAS analysis 1.25 g of the tellurium powder was weighed in a quartz flask of 50 ml and dissolved

**Table 2.** Experimental spark parameters

Spark Voltage (kV)	48
Repetition frequency (kHz)	1
Pulse length (μs)	20
Accelerating voltage (kV)	27
Main slit width (μm)	25
α-slit width (mm)	0.8
β-slit width (mm)	1.0

**Table 3.** List of interferences occurring in the SSMS Te spectra. The line free from interference corresponding to this element is also given

Element	Isotope	Interference	Line free from interference
Mg	24	$^{120}\text{Te}^{5+}$	$^{25}\text{Mg}^{2+}$
	25	$^{125}\text{Te}^{5+}$	
	26	$^{130}\text{Te}^{5+}$	
P	31	$^{124}\text{Te}^{4+}$	$^{31}\text{P}^{3+}$
Cu	63	$^{126}\text{Te}^{2+}$	—
	65	$^{130}\text{Te}^{2+}$	
Sb	121	$^{120}\text{TeH}^{1+}$	$^{121}\text{Sb}^{2+}$
	123	$^{123}\text{Te}^{1+}$	
I	127	$^{126}\text{TeH}^{1+}$	$^{127}\text{I}^{2+}$

in 20 ml HCl (sub-boiling) and 0.5 ml HNO<sub>3</sub> (sub-boiling). The volume was made up with high-purity water (Milli-Q water, 18 MΩ). For each sample 4 preparations and 4 blanks were measured.

The glow discharge mass spectrometer (GDMS) is of the type VG 9000. The sample to be analyzed forms the cathode of a low pressure discharge in an argon gas. The instrument has a double focusing mass spectrometer and ion detection is done on a Daly detector (low currents) or a Faraday bucket (high currents). Data acquisition and manipulation is performed on an Industry Standard IBM-XT computer. For GDMS analyses the Te powder was pressed into a bar of approximately 15 mm length and 2–3 mm width. The Te solid sample was cut into an electrode of approximately 15 mm length, 1–2 mm width, 1–2 mm thick. The sample was presputtered until a stable signal was obtained (ca. 30 min).

## Results

The SSMS spectrum of pure (6 N) tellurium shows many lines, a large percent of which are due to the matrix element that has 8 isotopes. As a consequence various interferences could be observed, the most important ones are summarized in Table 3. Of the elements typical for tellurium, only copper cannot be determined by SSMS. An analysis for phosphorus is possible based on the  $^{31}\text{P}^{3+}$  ion line, for magnesium on  $^{25}\text{Mg}^{2+}$ , for antimony on  $^{121}\text{Sb}^{2+}$  and for iodine on  $^{127}\text{I}^{2+}$ . Other elements, not mentioned in Table 3, which are interfered are Ba, La, Ce, Pr, Nd, Sm und Eu. These interferences are due to polyatomic ions, particularly tellurium oxide and tellurium hydroxide ions. Since all their isotopes are interfered, the determination of these elements can only be done based on doubly or triply charged ion lines. It should be

**Table 4.** Average RSC's obtained by SSMS for a tellurium matrix, <sup>a</sup> = RSC based on the doubly charged ion line

Element	RSC			
	Standard 1	Standard 2	Standard 3	<RSC>
Na	1.7 ± 0.4	2.3 ± 0.6	—	1.9 ± 0.3
Mg <sup>a</sup>	0.30 ± 0.14	0.58 ± 0.24	—	0.37 ± 0.12
K	13 ± 10	12 ± 6	—	12 ± 5
Ca	3.9 ± 2.1	12.0 ± 4.9	—	5.2 ± 1.9
Cr	1.2 ± 0.2	1.6 ± 0.2	1.1 ± 0.2	1.30 ± 0.12
Mn	1.5 ± 0.3	1.1 ± 0.1	—	1.14 ± 0.09
Fe	1	1	1	1
Co	1.2 ± 0.2	1.2 ± 0.3	—	1.20 ± 0.17
Ni	1.3 ± 0.2	1.1 ± 0.2	1.5 ± 0.3	1.25 ± 0.13
Zn	0.54 ± 0.13	0.47 ± 0.14	—	0.51 ± 0.09
As	0.36 ± 0.15	—	—	0.36 ± 0.15
Se	0.11 ± 0.05	—	—	0.11 ± 0.05
Mo	1.0 ± 0.2	1.1 ± 0.2	—	1.05 ± 0.14
Ru	—	3.6 ± 1.0	—	3.6 ± 1.0
Rh	—	2.7 ± 0.6	—	2.7 ± 0.6
Pd	—	1.2 ± 0.2	—	1.2 ± 0.2
Ag	2.1 ± 0.5	2.5 ± 0.7	2.3 ± 0.6	2.3 ± 0.3
Cd	1.2 ± 0.6	—	—	1.2 ± 0.6
Sn	1.9 ± 0.2	1.8 ± 0.4	1.6 ± 0.4	1.83 ± 0.16
Sb <sup>a</sup>	0.65 ± 0.04	0.61 ± 0.09	0.72 ± 0.11	0.65 ± 0.03
Ir	—	0.85 ± 0.18	—	0.85 ± 0.18
Au	—	0.33 ± 0.07	—	0.33 ± 0.07
Pb	3.4 ± 0.8	3.3 ± 0.7	2.3 ± 0.6	2.9 ± 0.4
Bi	—	0.92 ± 0.21	—	0.92 ± 0.21

mentioned, however, that none of these elements are commonly present in Te.

Each of the 3 tellurium "standard" samples were analyzed 3 times by SSMS. For the calculation of the concentration an internal standard element is needed. The least abundant matrix isotope <sup>120</sup>Te is not suitable for this purpose since its line blackening can only be measured at the lowest exposure for which the coulometer readings are unreliable. Therefore, Fe was selected as the internal standard element. Various exposures on a photoplate showed that this element was homogeneously distributed in the matrix, furthermore Fe is present at a concentration which can accurately be determined by AAS. Using the concentrations obtained by SSMS and AAS, the RSC could be determined according to:

$$\text{RSC}(x/\text{Fe})_{\text{Te}} = (C_x/C_{\text{Fe}})_{\text{SSMS}} / (C_x/C_{\text{Fe}})_{\text{AAS}} \quad (1)$$

The RSC's apply for an impurity element *x* relative to the internal standard element Fe in the matrix Te. The SSMS concentrations used in Eq. (1) were corrected for variations in detector mass response. The RSC's were averaged and the standard deviations were calculated for each standard. The weighted average of these RSC's and the corresponding uncertainties (standard deviations) were also calculated. The mean precision of the measurements was 22% relative standard deviation. These results are summarized in Table 4 for 24 elements. Especially for Al, Si and In irreproducible results were obtained due to their heterogeneous distribution in the sample and therefore these elements were not considered for RSC determination. It appears from these results that the RSC values for most elements are within the range 0.3–3, which is typical for SSMS. As could be expected, higher RSC values were found for the elements with low ionization potential such as K and Ca, and to a lesser extent

for Na; a lower RSC value was obtained for the volatile element Se. It is supposed that these RSC values, determined on pressed Te powders, also apply for solid Te samples. An analysis of a 6N Te powder after pressing it into compact electrodes showed that less than 1% of impurities (C, N, O) are present. Usually at this impurity level no matrix effects are observed. Using the RSC values obtained in this study, a 4N Te sample (powder) was analyzed versus Fe, of which the concentration was determined by GFAAS, as the internal standard element for the SSMS calculations. The same sample was also analyzed by GFAAS and GDMS. The results of these comparative analyses are shown in Table 5 for selected elements. The GDMS results were not corrected for differences in relative sensitivity among the elements. Only for K a large difference was found between GDMS and the other techniques. For GDMS an RSC for K comparable to the one applied for SSMS should be used. The high GDMS Al value is probably due to the sample preparation method where an Al holder was used for pressing the sample. For SSMS a good agreement was found with GFAAS, most results were within a factor of 1.5, when correcting for relative sensitivities.

For the analysis of high purity tellurium samples, it proved to be difficult to obtain an internal standard element for SSMS. Table 6, for instance, shows the results of the analysis of a 6N solid Te sample. By GFAAS only detection limits could be obtained whereas by SSMS circa 10 impurities were detected using a highest exposure of 600 nC. In such cases GFAAS can still be used for calibration when applied in combination with enrichment techniques such as described by Baranova et al. [10] (i.e. matrix separation by distillation or extraction of impurities). Another alternative is to use other sensitive quantitative techniques, e.g. activation analysis. If these possibilities are not applicable,

**Table 5.** Concentrations (ppmw) obtained by SSMS, GDMS and GFAAS for a 4N Te sample, <sup>a</sup> = corrected for RSC, I = interference

Element	Concentration (ppmw)		
	SSMS	GDMS	GFAAS
Be	<0.001	—	<0.01
C	≤0.08	≤50	—
N	≤0.05	≤0.09	—
Na	0.43 <sup>a</sup>	0.92	0.28
Mg	0.11 <sup>a</sup>	0.083	0.07
Al	0.11	2.3	0.15
Si	0.034	0.14	<0.5
K	0.035 <sup>a</sup>	0.50	≤0.03
Ca	0.11 <sup>a</sup>	<0.3	0.12
Ti	<0.009	—	<0.05
V	0.009	—	<0.03
Cr	0.85 <sup>a</sup>	1.1	0.88
Mn	0.13 <sup>a</sup>	<0.006	0.11
Fe	7.5 <sup>a</sup>	7.5	7.5
Co	0.016 <sup>a</sup>	0.018	<0.01
Ni	2.5 <sup>a</sup>	1.8	2.6
Cu	I	0.47	2.3
Zn	0.18 <sup>a</sup>	0.05	0.21
Ga	—	<0.02	<0.2
As	0.081 <sup>a</sup>	0.033	—
Se	1.9 <sup>a</sup>	1.4	2.7
Mo	0.10 <sup>a</sup>	0.14	≤0.1
Ru	0.060 <sup>a</sup>	0.078	0.04
Rh	2.6 <sup>a</sup>	1.5	2.8
Pd	1.4 <sup>a</sup>	0.69	1.1
Ag	23 <sup>a</sup>	5.3	18
Cd	0.044 <sup>a</sup>	<0.09	<0.02
In	0.40	0.14	0.20
Sn	0.27 <sup>a</sup>	0.67	0.25
Sb	4.9 <sup>a</sup>	5.0	5.8
Ir	0.18 <sup>a</sup>	0.072	<0.1
Pt	0.069	<0.07	0.09
Au	11 <sup>a</sup>	3.9	9.1
Pb	1.4 <sup>a</sup>	1.8	1.5
Bi	0.12 <sup>a</sup>	0.092	<0.1

quantitative results can still be obtained by SSMS by recording the spectra of the sample and a standard on the same photoplate. The SSMS result for Fe in Table 6 was obtained in this way. The SSMS detection limits, given in Table 7, were obtained after visual estimation of the blackenings on the photoplate. Most elements can be determined with limits of detection better than 10 ppb. The SSMS results were corrected with the RSC values obtained in Table 4. The same sample was also analyzed by GDMS, the results for some elements are also indicated in Table 6. For GDMS analysis the <sup>120</sup>Te matrix isotope was used as the internal standard. This possibility of GDMS to use the matrix as the internal standard is an advantage over SSMS. Another advantage of GDMS over SSMS is the speed of analysis. The GDMS analysis of the 4N sample and the 6N sample, of which the results are shown in Tables 5 and 6, took less than 3 h for each sample. The SSMS analysis for each sample took circa 12 h. On the other hand the SSMS sample preparation is very fast with a minimum risk of contamination, whereas for GDMS special sample shaping (cutting) was required. Surface contamination which could be introduced was afterwards removed by presputtering in the GDMS ion source.

**Table 6.** Results (ppmw) obtained by SSMS, GDMS and GFAAS for a 6N Te sample, <sup>a</sup> = corrected for RSC, I = interference

Element	Concentration (ppmw)		
	SSMS	GDMS	GFAAS
Li	<0.001	<0.003	<0.05
Be	<0.001	—	<0.01
B	<0.001	<0.003	—
C	≤0.02	≤1.8	—
N	≤0.005	≤0.1	—
O	≤0.05	≤0.6	—
F	<0.001	—	—
Na	0.11 <sup>a</sup>	<0.003	<0.05
Mg	<0.002 <sup>a</sup>	<0.004	<0.02
Al	0.008	<0.004	<0.05
Si	0.02	0.02	<0.3
P	<0.002	<0.004	—
S	<0.005	<0.03	—
Cl	<0.001	—	—
K	<0.003 <sup>a</sup>	<0.050	<0.05
Ca	0.018 <sup>a</sup>	<0.23	<0.05
Sc	<0.001	—	—
Ti	<0.001	≤0.03	<0.05
V	<0.001	<0.003	—
Cr	0.011 <sup>a</sup>	<0.008	<0.02
Mn	0.008 <sup>a</sup>	<0.007	<0.02
Fe	0.012 <sup>a</sup>	<0.009	<0.05
Co	<0.004 <sup>a</sup>	<0.007	<0.02
Ni	0.025 <sup>a</sup>	<0.03	≤0.02
Cu	I	<0.003	<0.05
Zn	<0.004 <sup>a</sup>	<0.004	<0.05
Ga	≤0.03	<0.015	<0.1
Ge	<0.002	<0.008	—
As	<0.002 <sup>a</sup>	<0.02	<0.1
Se	<0.015 <sup>a</sup>	—	—
Br	<0.002	<0.009	—
Rb	<0.001	<0.01	—
Sr	<0.001	<0.006	—
Y	<0.001	—	—
Zr	<0.002	—	—
Nb	<0.001	<0.002	—
Mo	<0.005 <sup>a</sup>	<0.05	<0.1
Ru	<0.001 <sup>a</sup>	<0.09	—
Rh	<0.001 <sup>a</sup>	—	—
Pd	<0.004 <sup>a</sup>	<0.07	<0.05
Ag	<0.001 <sup>a</sup>	—	<0.01
Cd	0.018 <sup>a</sup>	<0.05	<0.02
In	0.017	<0.02	<0.06
Sn	<0.003 <sup>a</sup>	<0.05	<0.05
Sb	<0.01 <sup>a</sup>	<0.04	<0.05
I	<0.005	—	—
Cs	<0.002	—	—
Ba	<0.02	—	—
Hf	<0.008	—	—
W	<0.009	—	—
Re	<0.005	<0.006	—
Os	<0.007	—	—
Ir	<0.006 <sup>a</sup>	—	—
Pt	<0.008	<0.06	<0.05
Au	<0.025 <sup>a</sup>	<0.05	<0.01
Hg	<0.01	—	—
Tl	<0.005	—	—
Pb	<0.002 <sup>a</sup>	<0.009	<0.05
Bi	<0.004 <sup>a</sup>	<0.007	<0.05
Th	<0.004	<0.007	—
U	<0.004	<0.007	—

**Table 7.** Detection limits obtained by SSMS and GFAAS for the tellurium matrix, <sup>a</sup> = GFAAS measurement after extraction method

Element	Detection limit (ppba)	
	SSMS	GFAAS
Li	0.4	50
Be	0.4	10
B	0.6	—
C	0.5	—
N	0.5	—
O	0.6	—
F	0.6	—
Na	0.4	50
Mg	19	20
Al	0.8	50
Si	0.8	300
P	7.0	—
S	20	—
Cl	1.0	—
K	0.08	50
Ca	0.1	50
Sc	1.0	—
Ti	1.0	50
V	1.0	—
Cr	0.8	20
Mn	0.9	20
Fe	1.0	50
Co	0.8	20
Ni	1.6	20
Cu	I	50
Zn	7.5	50
Ga	2.0	100
Ge	3.0	—
As	3.0	100
Se	27	20 <sup>a</sup>
Br	3.0	—
Rb	2.0	—
Sr	2.0	—
Y	1.0	—
Zr	3.0	—
Nb	1.0	—
Mo	6.0	100
Ru	1.0	50
Rh	0.6	50
Pd	4.0	50
Ag	1.0	10
Cd	4.0	20
In	1.5	60
Sn	4.0	50
Sb	1.0	50
I	5.0	—
Cs	2.0	—
Ba	20	—
Hf	6.0	—
W	6.5	—
Re	3.0	—
Os	5.0	—
Ir	3.5	500
Pt	6.0	50
Au	18	10
Hg	7.0	—
Tl	3.0	—
Pb	1.0	50
Bi	2.0	50
Th	2.0	—
U	2.0	—

Although the detection limits, shown in Table 6, are not the ultimate GDMS detection limits, SSMS detection limits for various elements in Te are at present better than those obtainable by GDMS (e.g. C, N, O, S, Ca and others). GFAAS analysis yields accurate results but the determination of this large number of elements is very time consuming (> 1 week).

### Conclusion

It was possible to determine RSC values for 24 elements in tellurium after SSMS and GFAAS analyses of 3 in-house "standard" samples. Most of the RSC values lie in the range 0.3–3, which is typical for SSMS.

A 4N Te sample was analyzed by various techniques namely SSMS, GFAAS and GDMS. In general, the results agreed reasonably well considering that for GDMS preliminary results were used without RSC correction. With RSC correction SSMS results agreed with GFAAS results within a factor of 1.5.

A high purity Te sample (6N) was analyzed by SSMS, GDMS and GFAAS. By GFAAS only detection limits could be obtained, while SSMS and GDMS proved to be competitive techniques for high-purity samples. Each technique is characterized by several disadvantages and advantages like e.g. for GDMS the possibility to use the matrix element as the internal standard and the speed of analysis, for SSMS the fast sample preparation with a lower risk of contamination. Further research is required to study the possibilities of GDMS compared to SSMS for high purity samples. We also intend to check whether it is possible to significantly shorten the SSMS analysis time by modifying the spark circuit and the extraction ion optics of the mass spectrograph, as proposed by Sikharulidze [11].

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