Innovative pocket-sized analyzers for continuous monitoring of volatile bio-marker molecules exhaled via breathing and skin

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Continuous personal medical monitoring task – challenge for doctors and engineers of 21-st century !

- Permanent metabolism diagnostics and control;
- Control of drugs therapeutic effect;
- Early diagnostics on the basis of long term data files (molecular life stories);
- Personal health and good aging predictive analysis and adjustments.

Continuous monitoring! Good idea! But how to do?

Each 15 minutes – to make a blood test?

**Every hour to go for a probing at the laboratory?**

To get electrodes implanted?

Volatile bio-marker molecules (VBMs) detection in gaseous products excreted via breathing and skin -

a possible way for non-invasive medical diagnostics !

Our cars are already controlled with gas analyzers of exhausted gases to form optimal fuel mixture and to achieve best engine performance mode….

And what about us, humans?
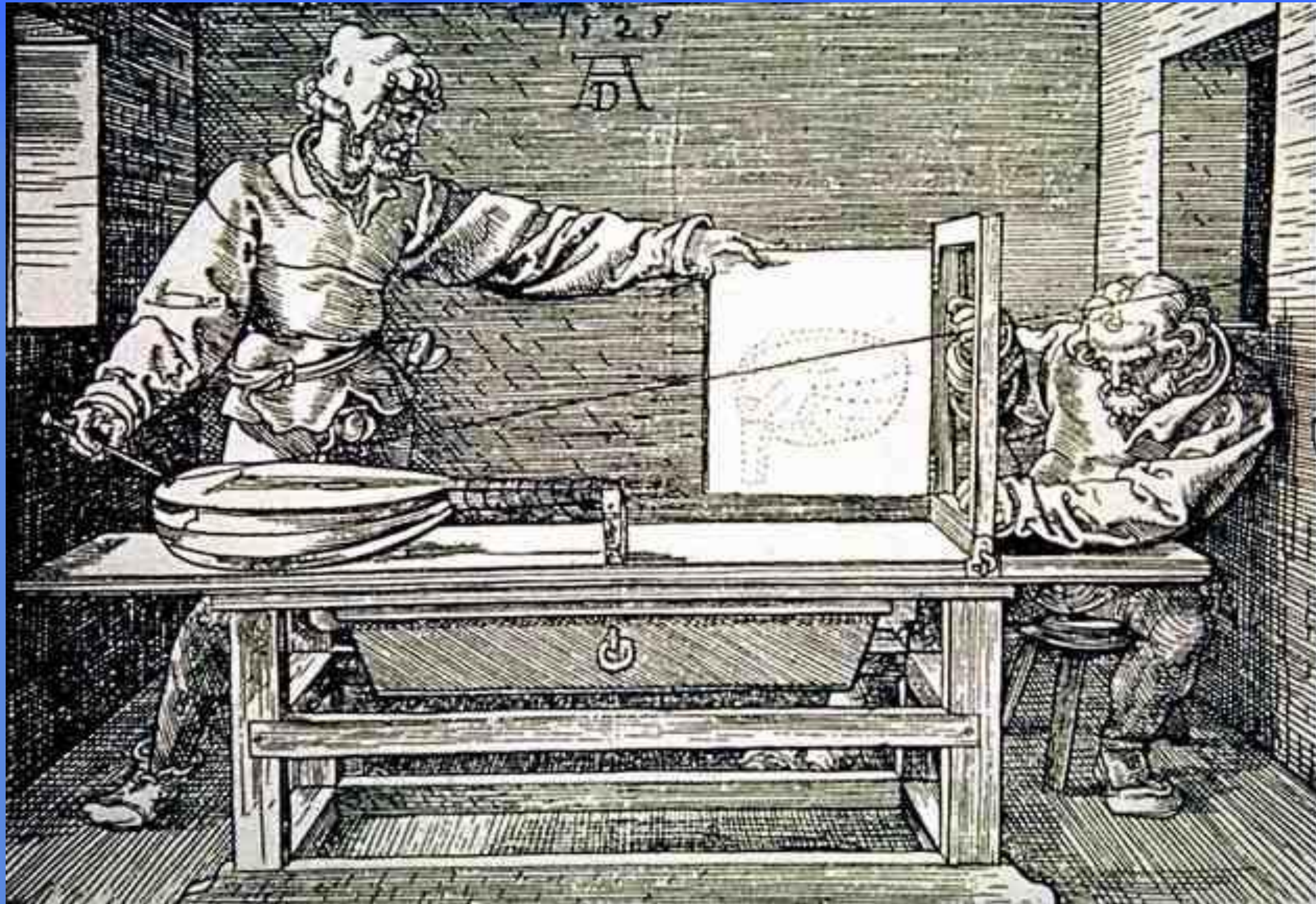
"Art of Measurement"
by Albrecht Durer

Mass-spectrometers ?
Gas chromatography ?
Fourier transform IR-spectrometers ?
Ion mobility spectrometers ?
Electro-chemical cells?
Thermo-catalytic detectors ?
IR laser diodes ?

For a personal continuous diagnostics it is necessary simultaniously :

Small dimensioins (pocket size);
Low weight (similar);
24 hours rechargeable battery supply;
Low price (<$1000);
Sufficient sensitivity and detector resolution.

Electron Spectroscopy concept

Traditional methods of electron spectroscopy (ES), which have been first discovered by A.Einstein (Nobel prize for the photo-effect explanation) and K.Zigban (Nobel prize for ESCA method - Electron Spectroscopy for Chemical Analysis), are one of the most informative means for chemical structure analysis of the matter (see S.Hufner, «Photoelectron Spectroscopy. Principles and Applications», Springer, 2003). According to ES, the identification of atoms and molecules is carried out by the energy analysis of characteristic electrons formed in ionization process of atoms or molecules A during collisions with particles B* of definite energy (photons, excited atoms, etc.)

$$A + B^* \rightarrow A^+ + B + e$$

On a measured energy E_e of characteristic electrons and known energy E_p of particles B* there may be determined the ionization potential E_i of the analyzed atoms or molecules A, as

$$E_i = E_p - E_e \qquad\qquad (1)$$

and, thus, the determination of a kind of atoms or molecules A can be done.

Traditional Electron Spectroscopy

However, significant deficiency of traditional ES method reveals in that it operates only in a high vacuum and expensive and sophisticated means for sample preparation and injection into vacuum are necessary for them. For this reason the electron spectroscopy didn't leave till now the walls of laboratories to enter wide analytical practice.

The reason is that the "heart" of classical electronic spectroscopy is an analyzer with high vacuum and of considerable geometrical size for a dispersion of electrons' motion in "momentum-coordinate" space (e.g. a cylindrical or spherical deflection mirror, an electrostatic or magnetic lens, time-of-flight tube, etc). Here electron takes off from a point of the birth with a certain momentum vector, then it is deflected in the field of the analyzer and reaches the detector's point, calculated according to the motion equations. Hence, the described electron energy analyzers actually measure electron's momentum on which its initial kinetic energy is recalculated. So, a rigid requirement arises to prevent electrons' angular scattering on residual gas particles inside of the energy analyzer, thus maintenance of high vacuum is necessary. However, to identify atoms or molecules according to Eq. (1), we need only the value of kinetic energy, instead of the momentum!

The way out is CES - Collisional Electron Spectroscopy (US patent 7,309,992 by A.Kudryavtsev, A.Tsyganov)

Collsional Electron Spectroscopy concept

It is based on the fact that in a single elastic collision with a gas particle the characteristic electron loses only a small part $\sim 2m/M_b < 10^{-4}$ from its initial kinetic energy (m – mass of electron, M_b – mass of a gas molecule). Therefore, if before energy measurement the electron will suffer, say, 100 elastic collisions, it will completely "lose" its initial direction of the momentum vector, but the relative loss of its kinetic energy δ_E will have rather small value < 1 %. We can spatially confine electron's path inside of a gas filled energy analyzer in such a way that only a limited number (from 100 to 1000) of collisions with gas particles occur till the electron being neutralized on the electrodes or the walls of the analyzer. In this case, electron's motion inside of the analyzer will be diffusive one and considerations for Brownian particle may be used. It provides the restriction that, at least, one of the geometrical dimensions L of the analyzer must be less than some tens of mean free paths of electron, if we want that collisional "degradation" of electron's initial characteristic energy doesn't exceed the pre-determined level δ_E.

$$L < \frac{\sqrt{\delta_E \cdot M_b / m}}{N_b \cdot \sigma_e} \qquad (2)$$

where N_b - gas concentration, σ_e – elastic cross-section of electron scattering on gas particles. For typical values of electron elastic cross-section σ_e the expression (2) can be written as $pL < 10$ cm*Torr (p - gas pressure in the detector), at the atmospheric pressure $L < 0.1$ mm.

Micro-plasma CES detector diagram
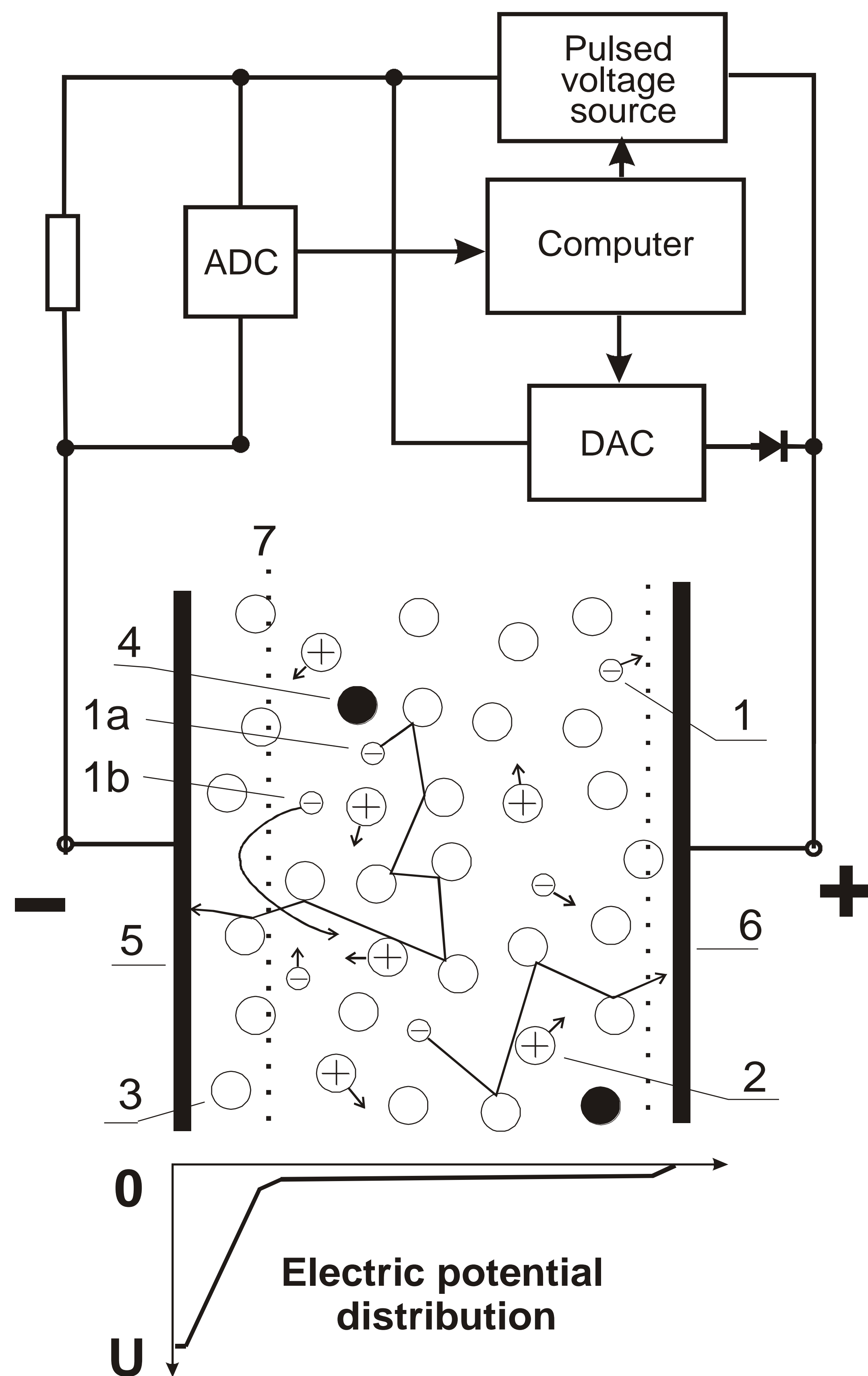
Depending on the applied voltage, the current between electrodes 5 and 6 is measured in a routine way, for example, from the voltage drop across resistor connected to this circuit. The voltage from the resistor must be low enough to avoid errors in the current-voltage characteristic of the detector, so it is amplified with conventional electronic circuits. Then the current-voltage characteristic is plotted. For example, the voltage from resistor is applied to the input of an amplifier with an analogue-digital converter (ADC) and further to computer for the data acquisition and processing. A digital-analogue converter (DAC) is also connected to the computer. The positive voltage at the DAC output is applied to anode 6 in the range of 0 to 25 V (the ionization threshold for helium atoms). Computer also controls pulsed power source for a glow discharge ignition to create an afterglow plasma. It is designed using high voltage transistor switch for a pulsed commutation of the DC voltage 200 – 600 V applied to the anode 6. The repetition rate of the pulses from said power unit, their duration and amplitude can also be controlled by the computer.

Microplasma CES detector
for continuous operation mode

СПОСОБ АНАЛИЗА ГАЗОВ И ИОНИЗАЦИОННЫЙ ДЕТЕКТОР
ДЛЯ ЕГО ОСУЩЕСТВЛЕНИЯ
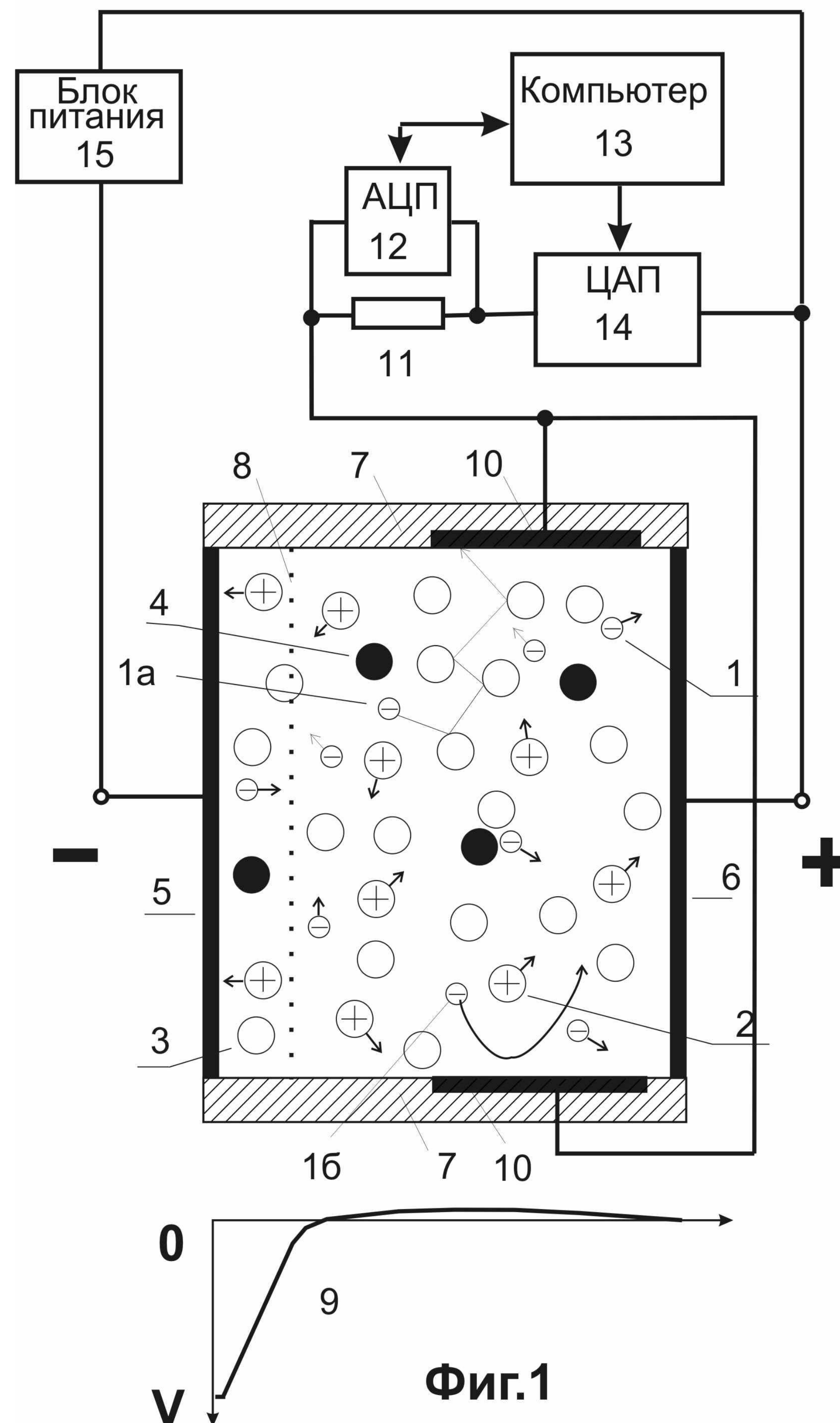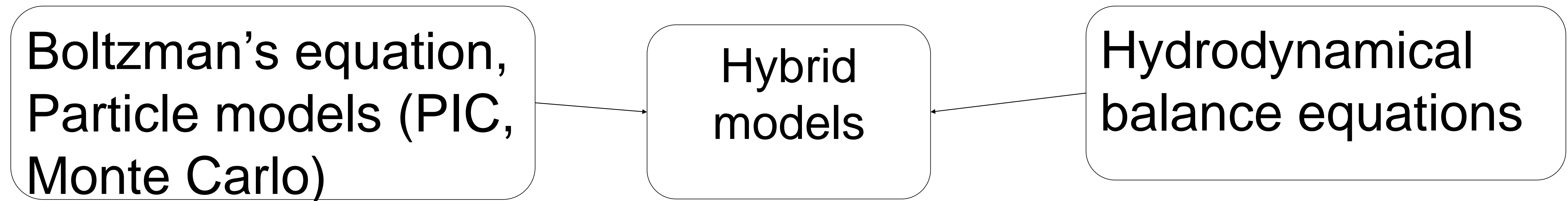
Фиг.1

Computational code for CES detector modelling

Kinetic approach

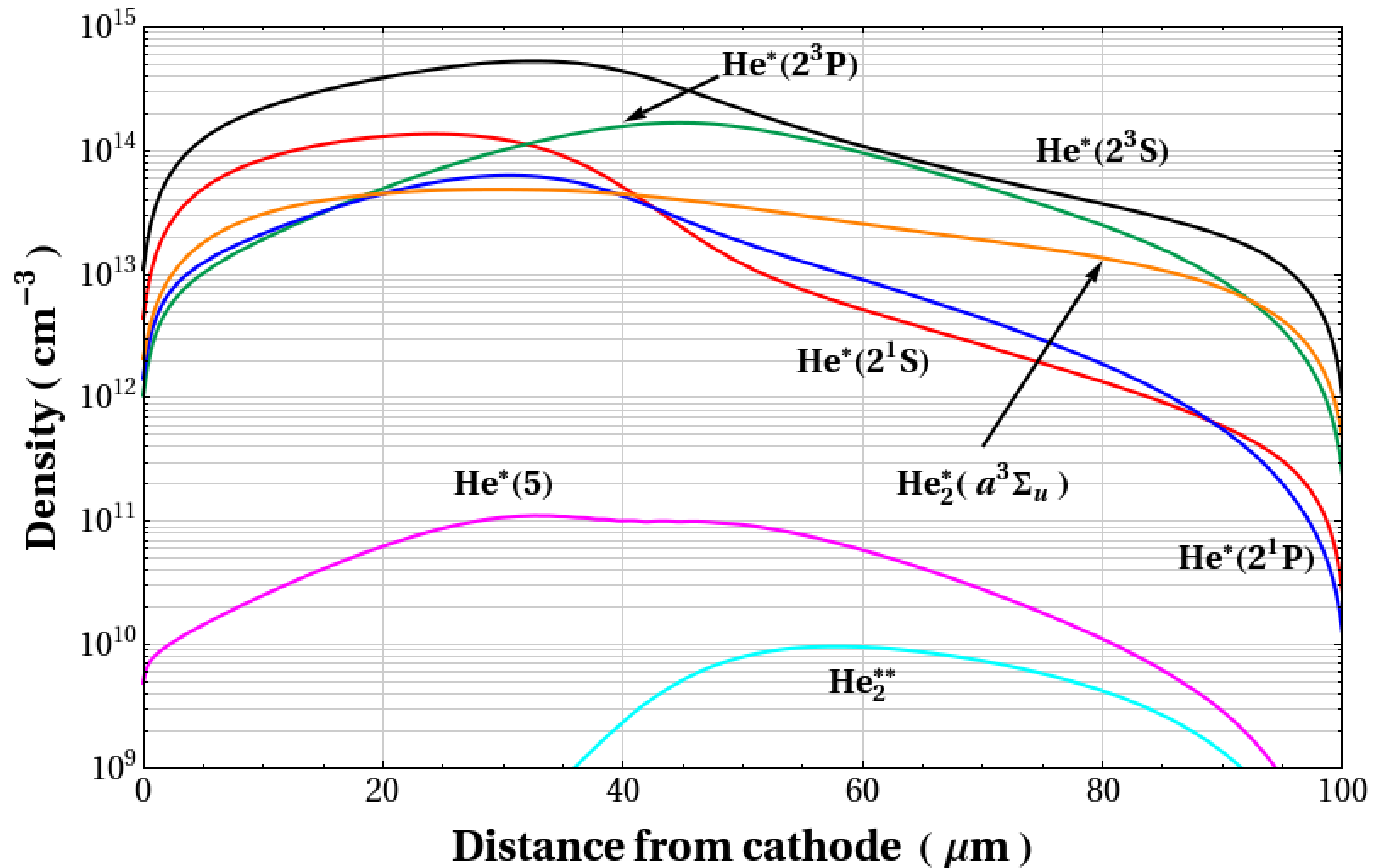Hydrodynamical approach

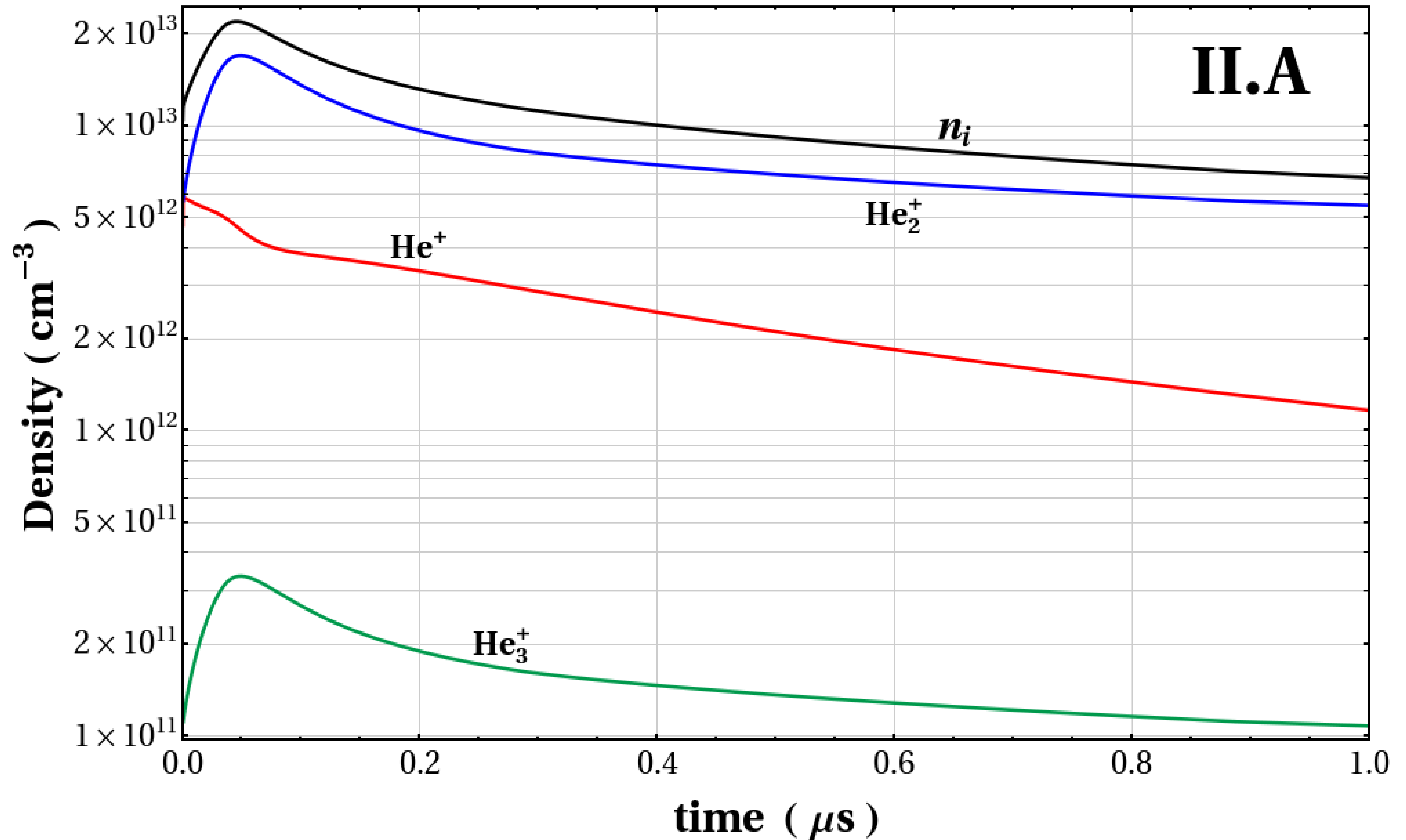Boltzman's equation, Particle models (PIC, Monte Carlo)

Hybrid models

Hydrodynamical balance equations

Spatial distribution of excited particles.

Time-dependent density profiles of charged particles at microplasma CES detector

Time-dependent density profiles of excited He atoms and molecules during first microsec of delay
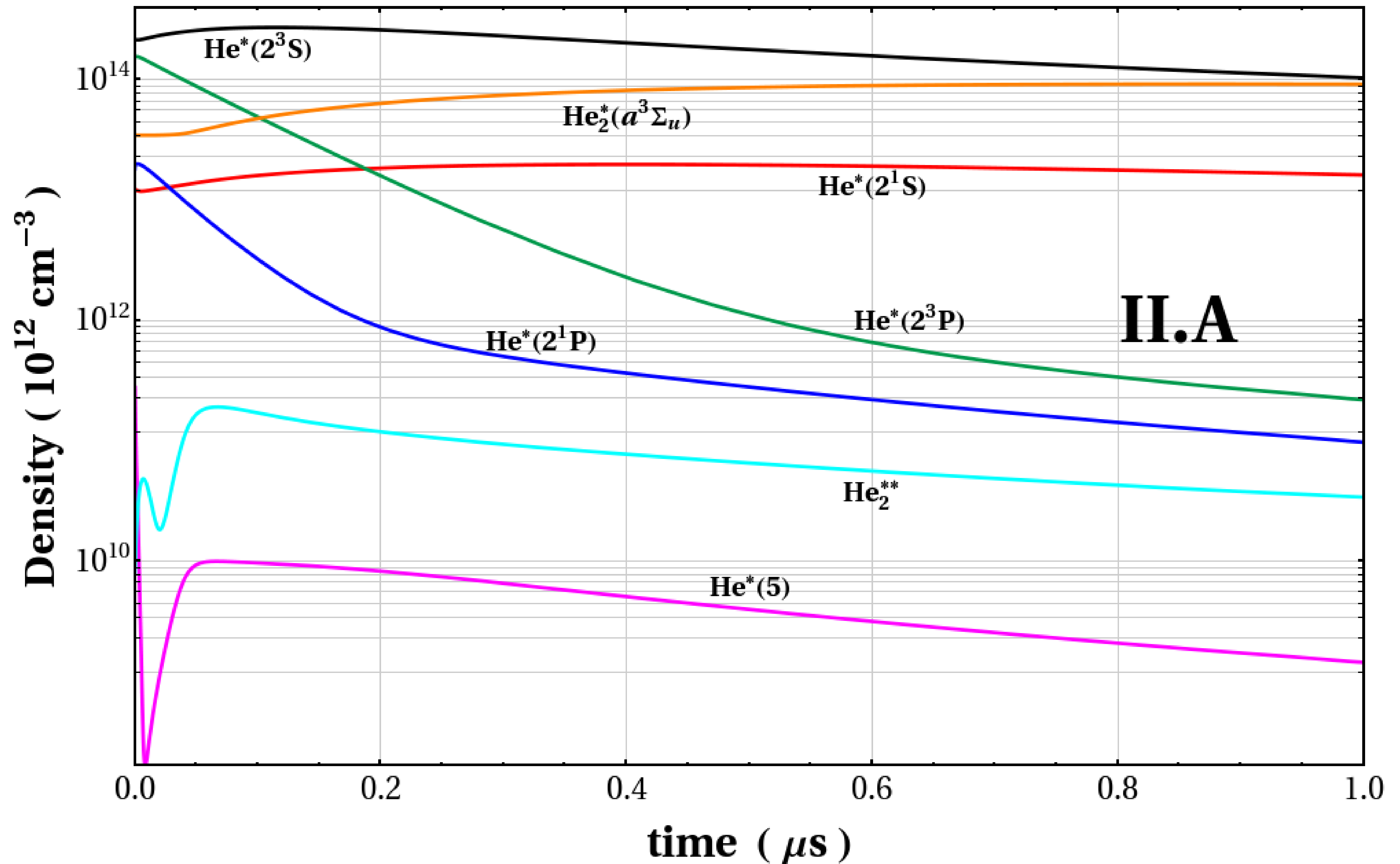
Electric field potential at inter-electrode gap during some moments of afterglow decay
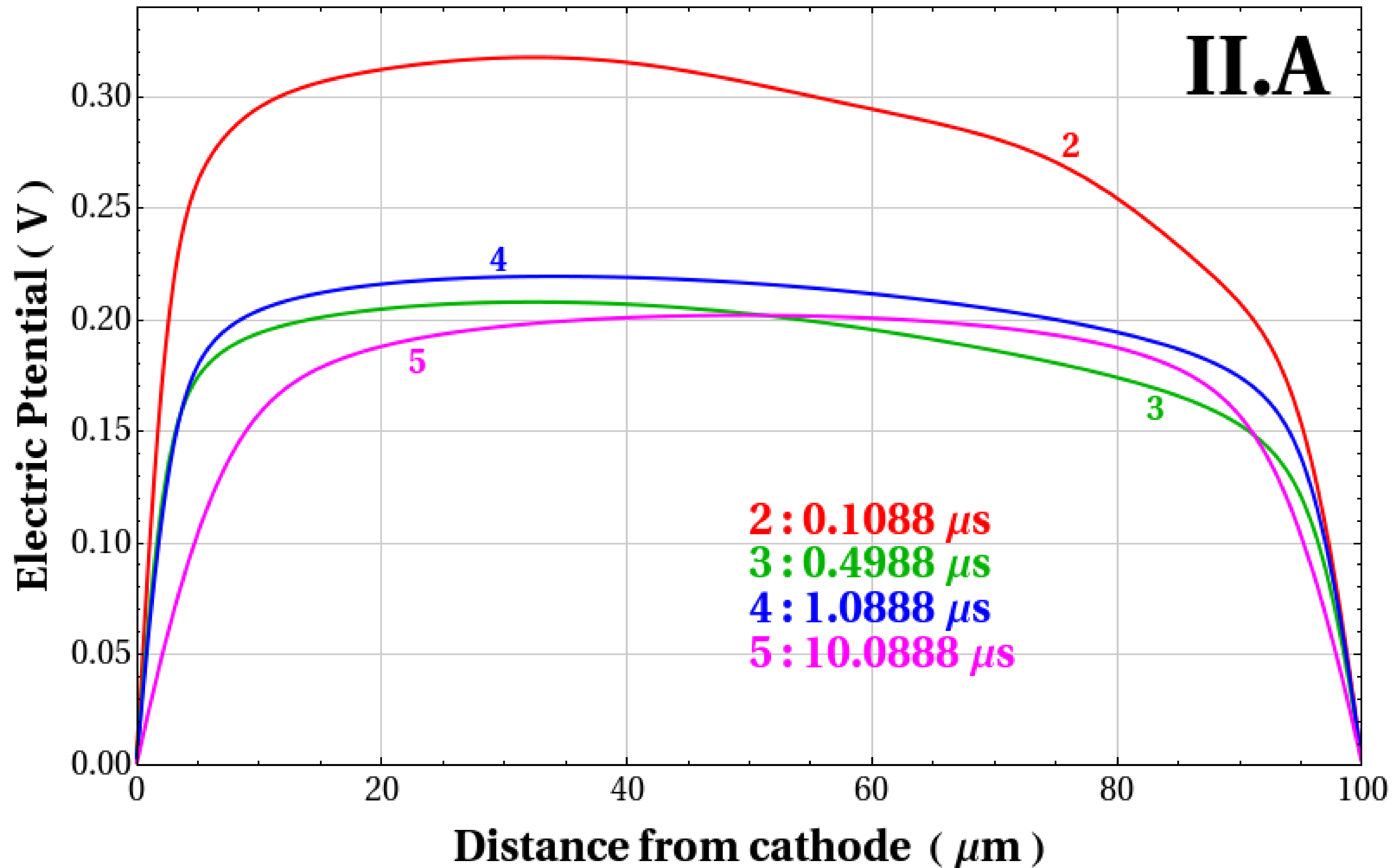
Electron and ion spatial distribution at inter-electrode gap during afterglow decay
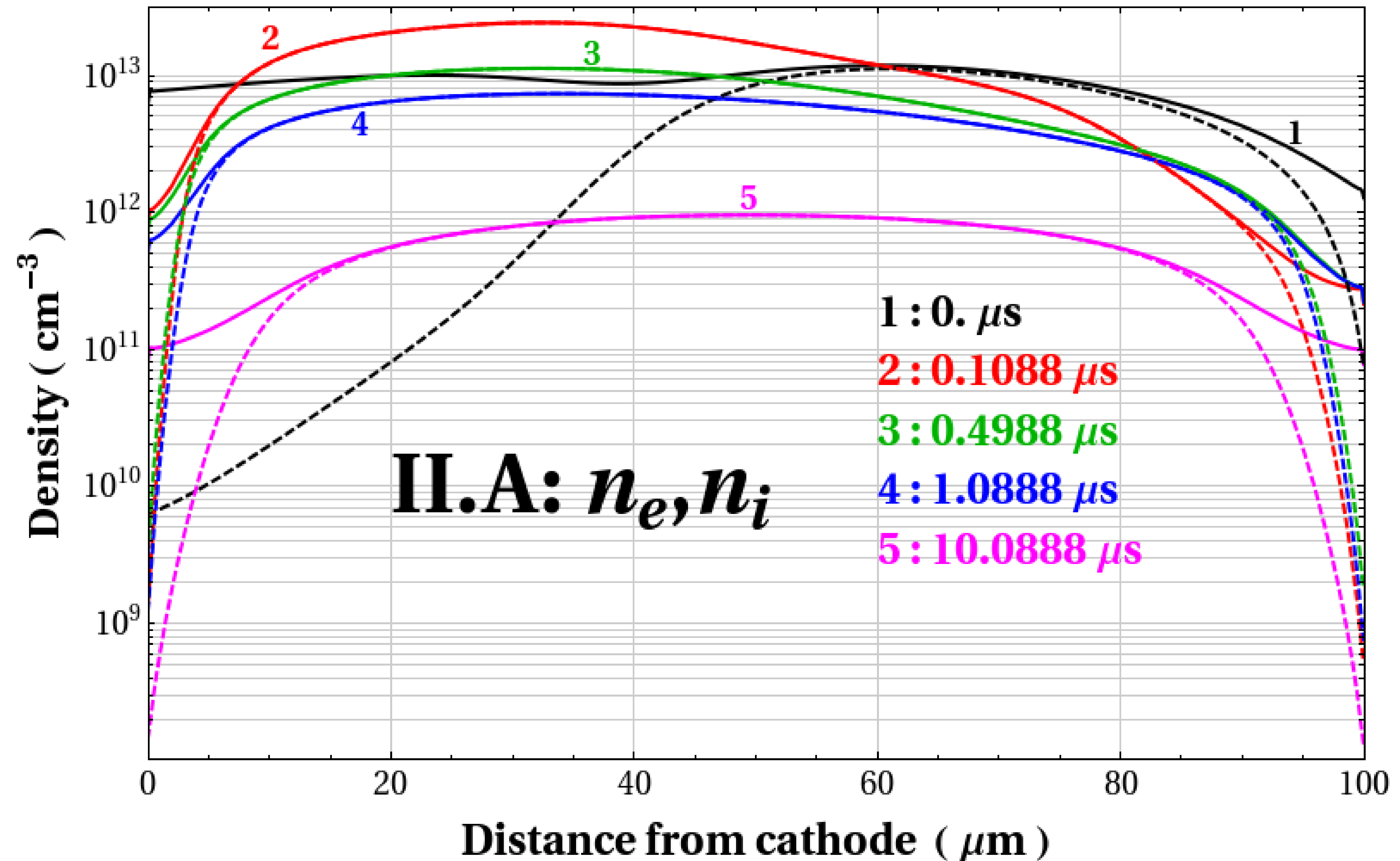
Spatial distribution of triplet He atoms at inter-electrode gap during afterglow decay
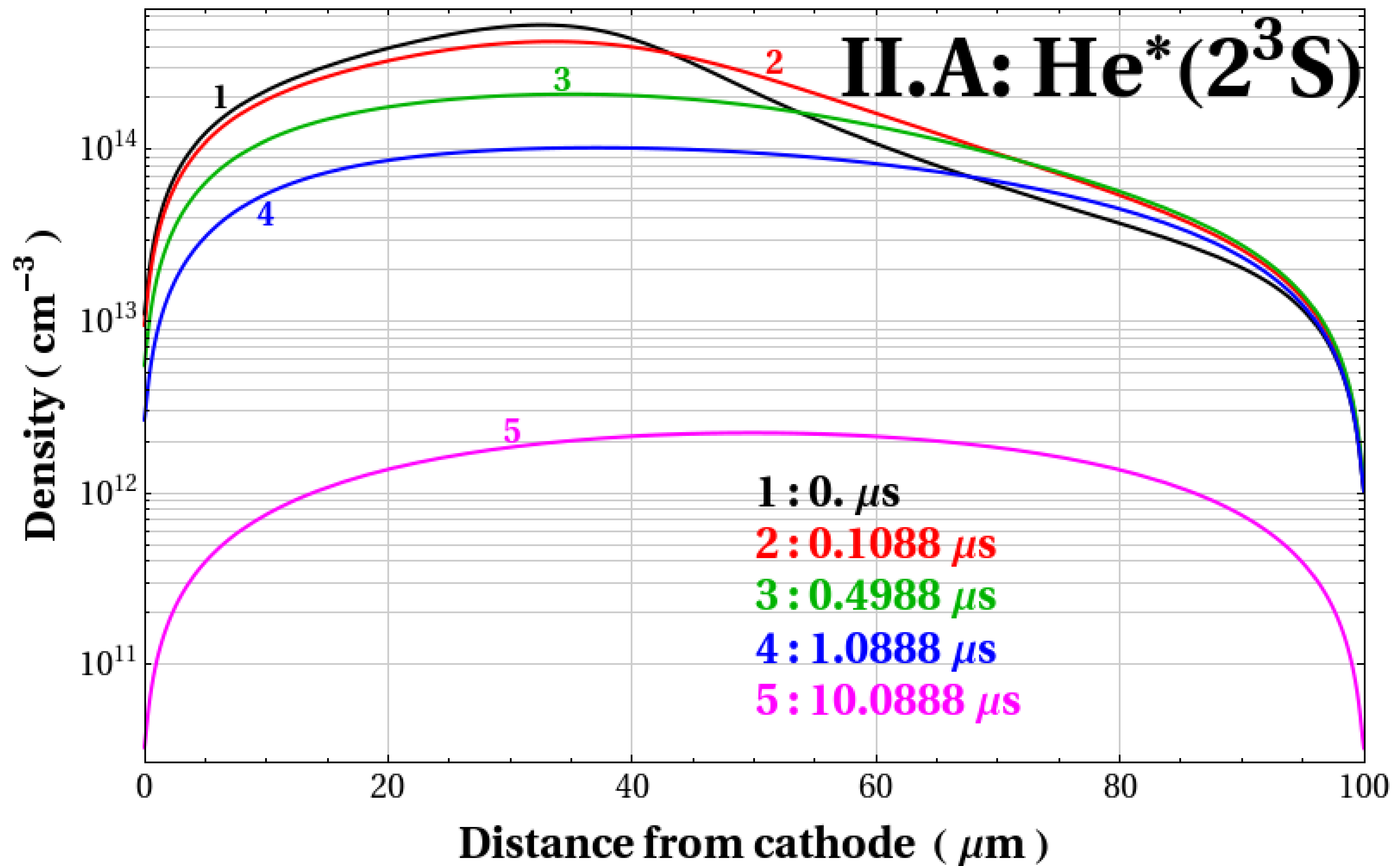
Spatial distribution of singlet He atoms at inter-electrode gap during afterglow decay
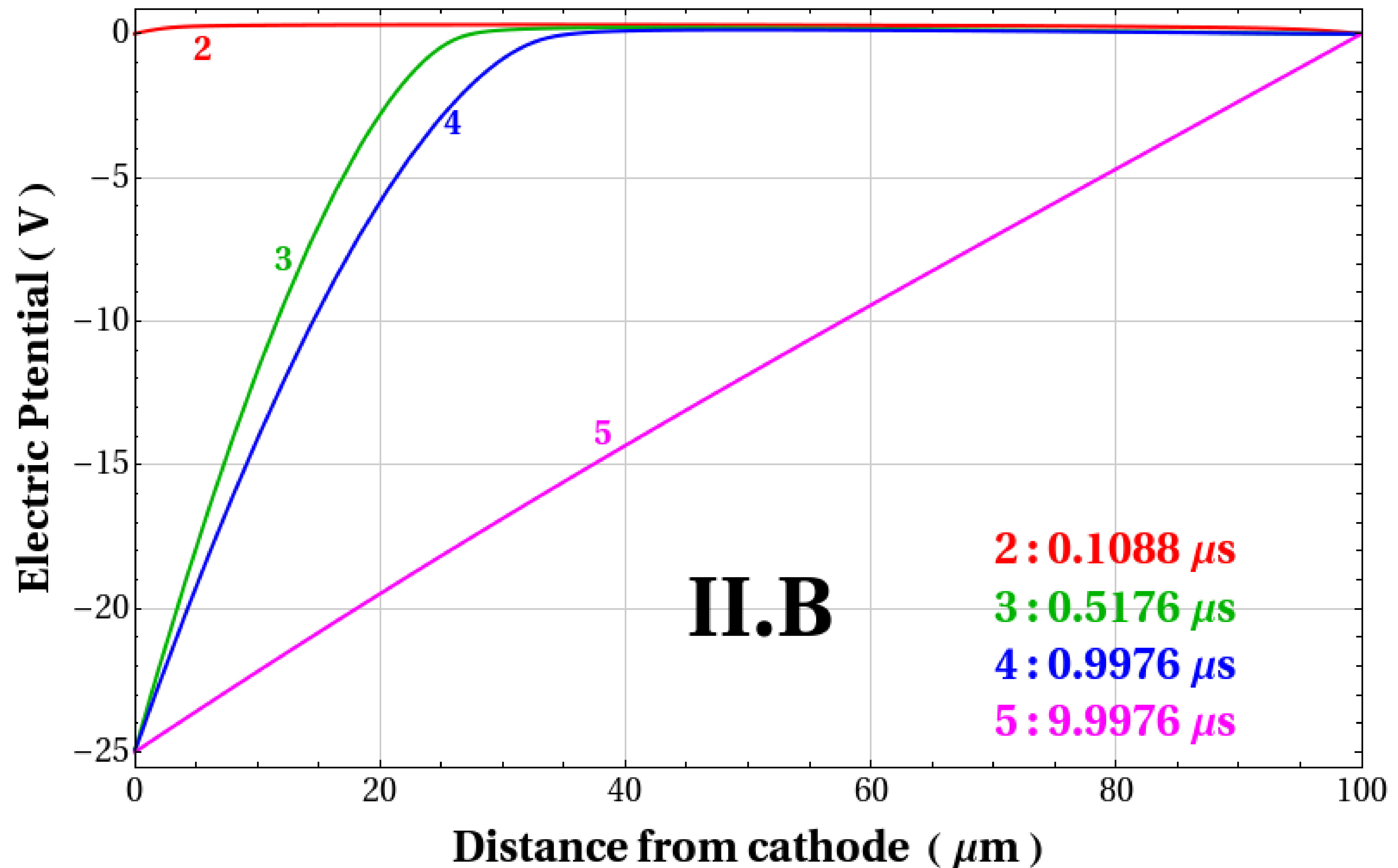
Micro-plasma CES detector
(microprocessor based control board)
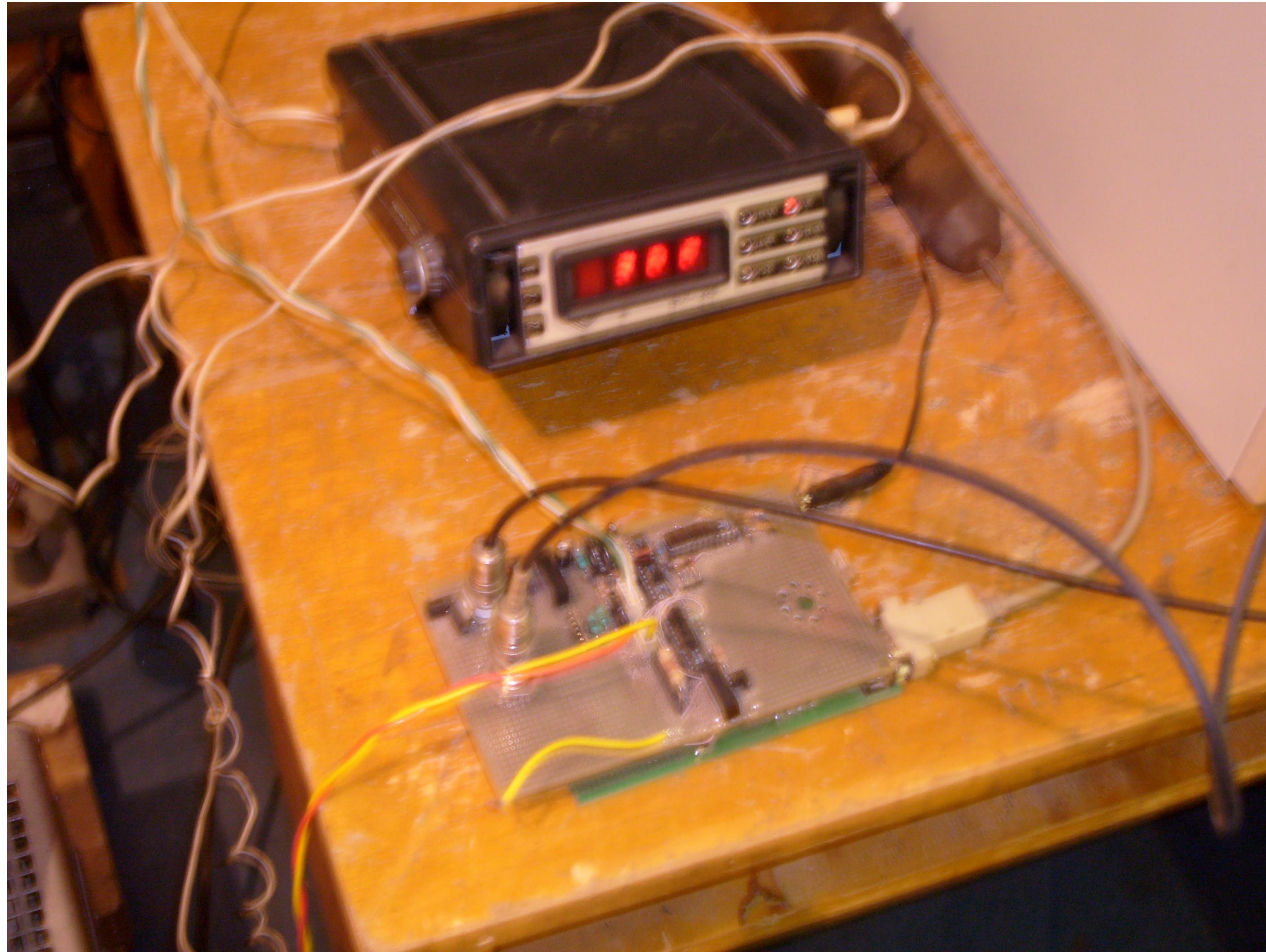
Electron energy spectrum at micro-plasma CES detector
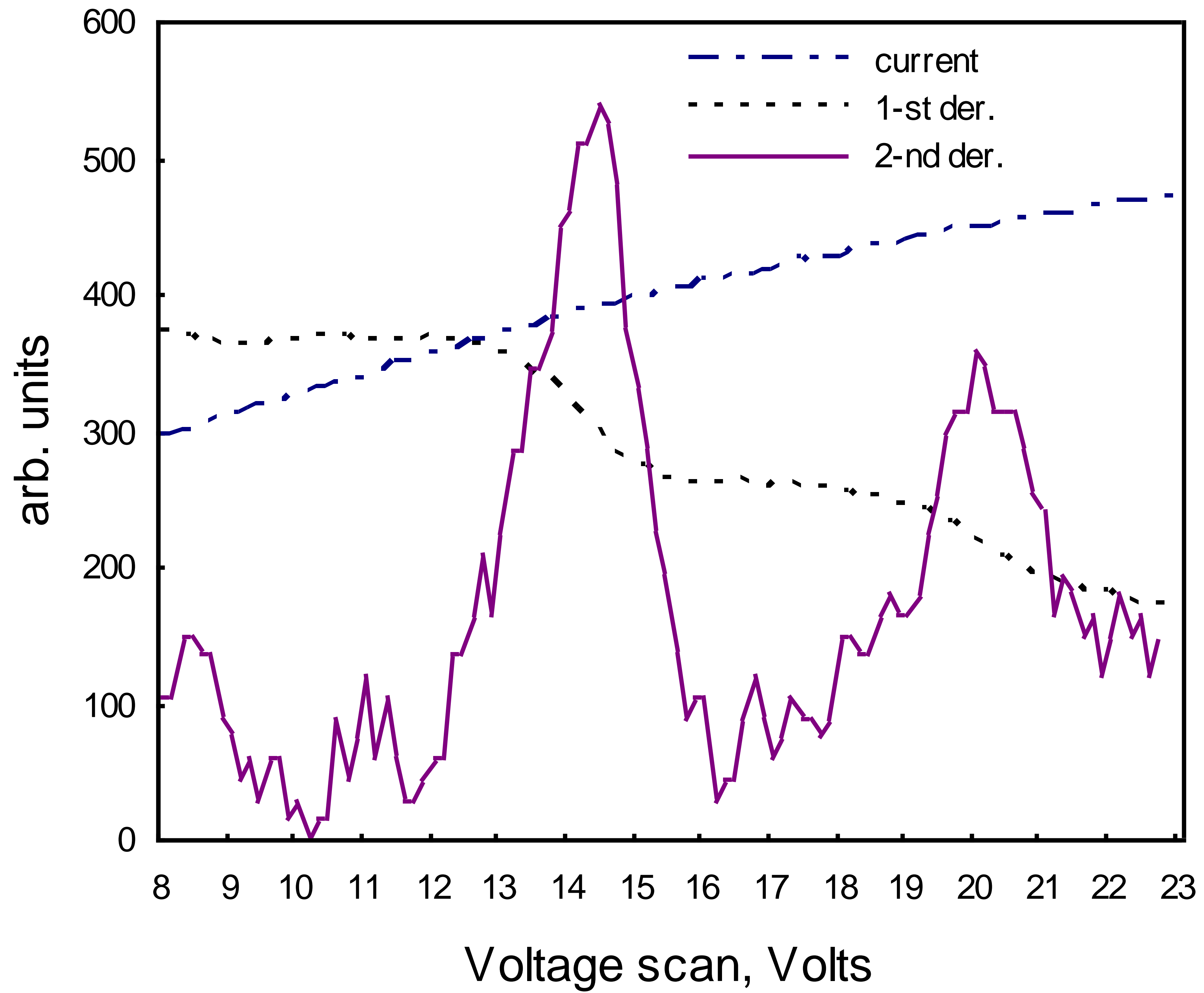
Current-voltage curve and electron energy spectrum at micro-plasma CES detector
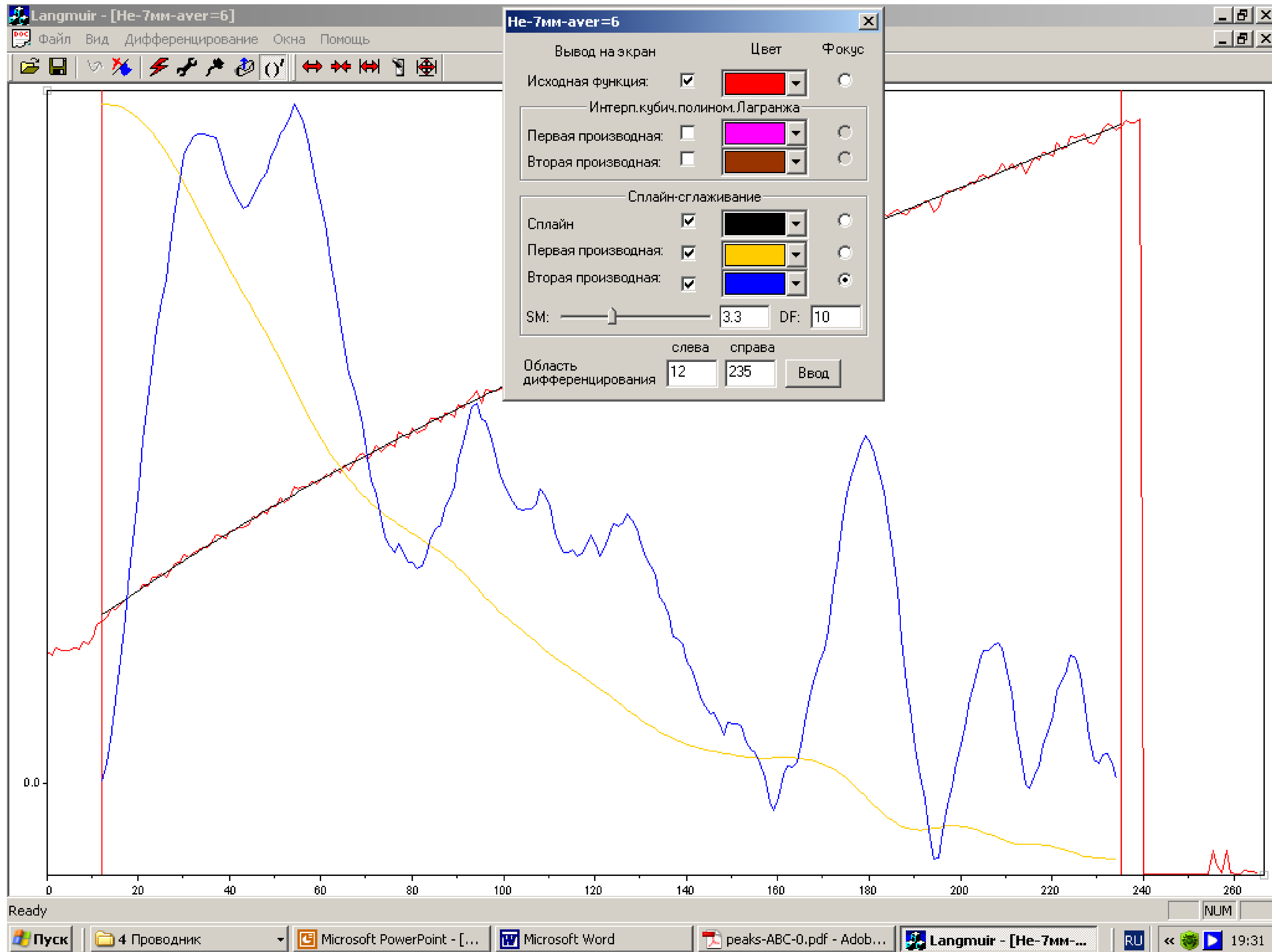
Current-voltage curve and electron energy spectrum at micro-plasma CES detector
P (He) = 40 Torr, L = 1 mm, t = 20 microsec
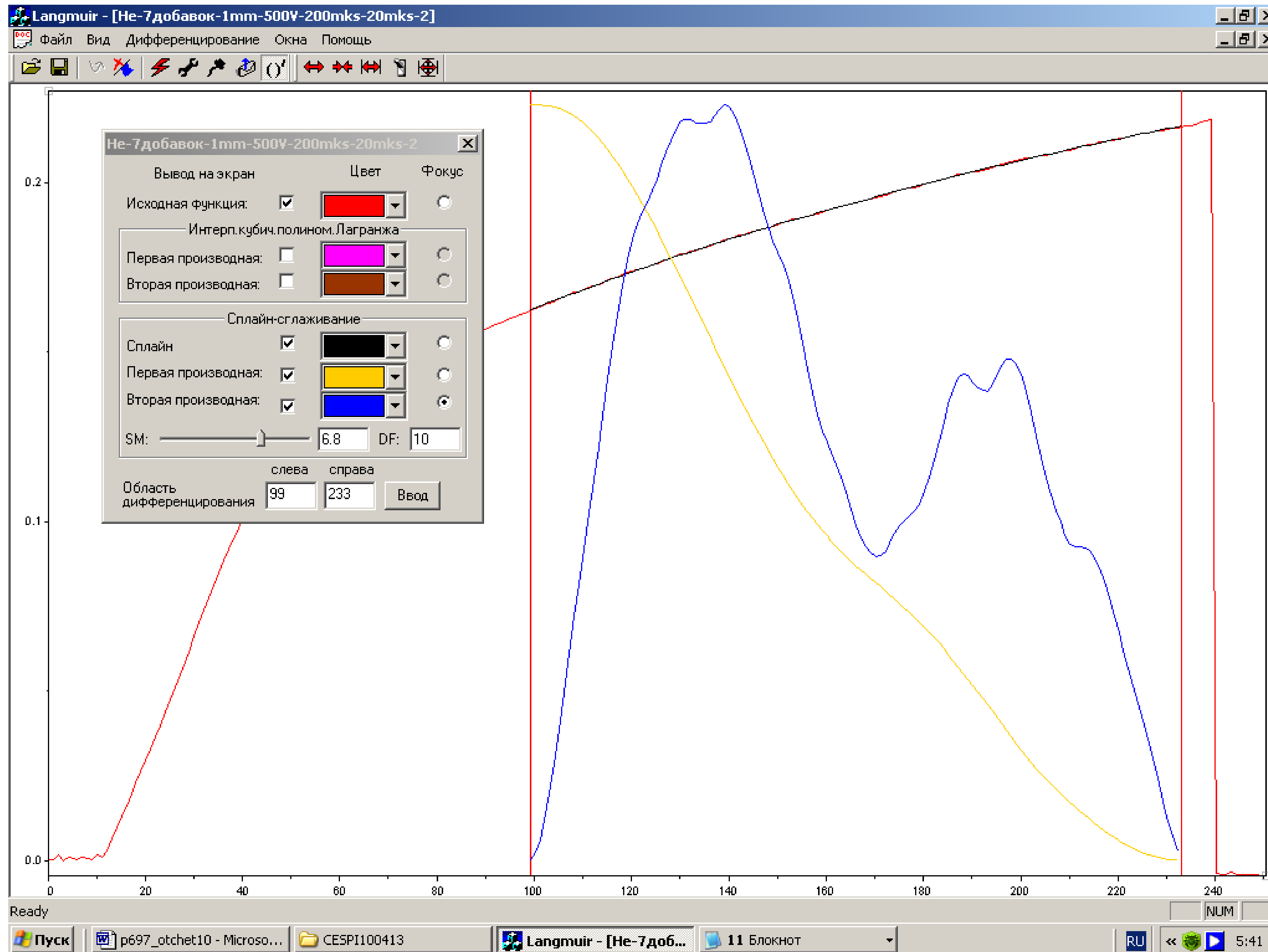
Diagram of VUV-photoionization CES detector
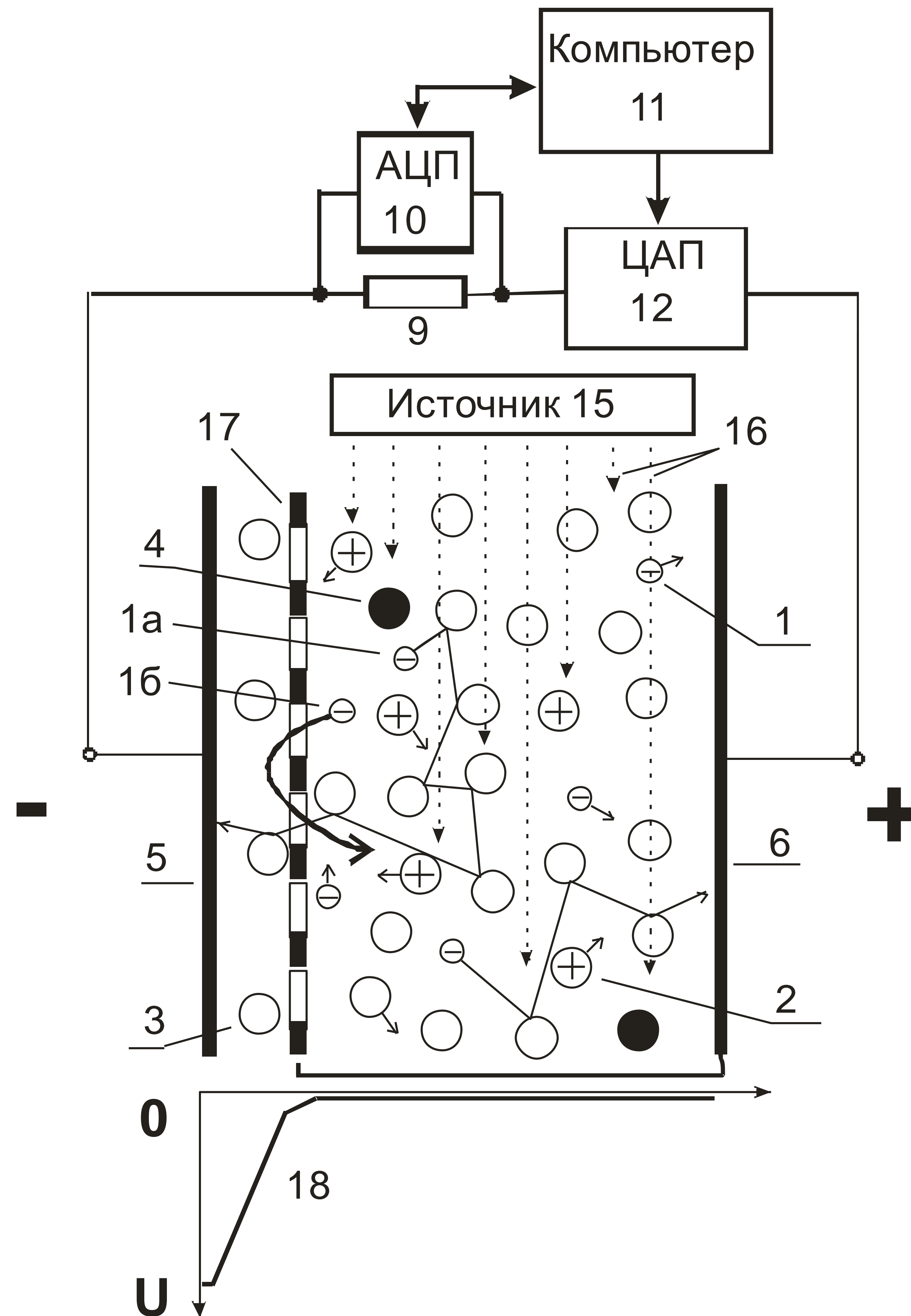
Diagram of VUV-photoionization CES detector of co-axial configuration

Electro energy spectrum at VUV-photoionization CES detector
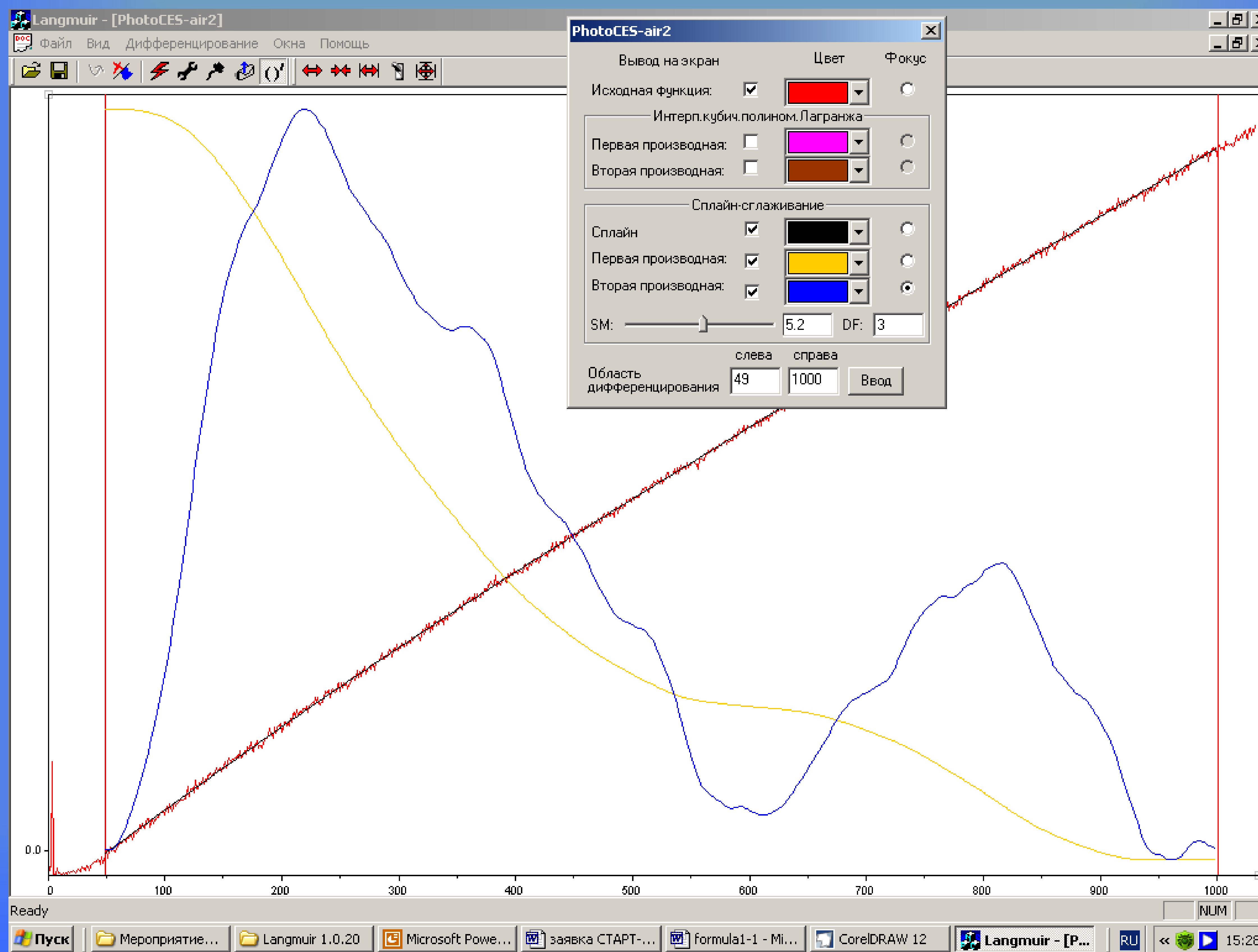
Experimental prototype of VUV-photoionization CES detector

List of detectable molecules

Chemical formula	Name	Ionization potential	
ClO_2	Chlorine dioxide	10.33	0.02
Cl_2S	Sulfur dichloride	9.45	0.03
HNO	Nitrosyl hydride	(10.1)	
HS	Mercapto	10.4219	0.0003
H_2S	Hydrogen sulfide	10.457	0.012
H_3N	Ammonia	10.070	0.019
H_4N_2	Hydrazine	8.1	0.14
Hg	Mercury	10.43749	
I_2	Iodine	9.3074	0.0000
NO	Nitric oxide	9.2643	0.00004
NO_2	Nitrogen dioxide	9.586	0.002
OS	Sulfur monoxide	10.294	0.004
Pb	Lead	7.41664	
PbS	Lead(II) sulfide	(8.5	0.5)
S_2	Disulfur	9.356	0.001
CH_2I_2	Diiodomethane	9.46	0.01
CH_2N_2	Diazomethane	8.999	0.000
CH_6N_2	Methylhydrazine	7.7	0.15
C_2H_4	Ethylene	10.5138	0.0005
$C_2H_4Br_2$	1,2-Dibromoethane	10.35	0.05
C_2H_6O	Ethanol	10.43	0.04
C_2H_6O	Dimethyl ether	10.025	0.024
C_3H_6O	Acetone	9.703	0.005
C_3H_8O	1-Propanol	10.18	0.05
C_3H_8O	2-Propanol	10.17	0.01

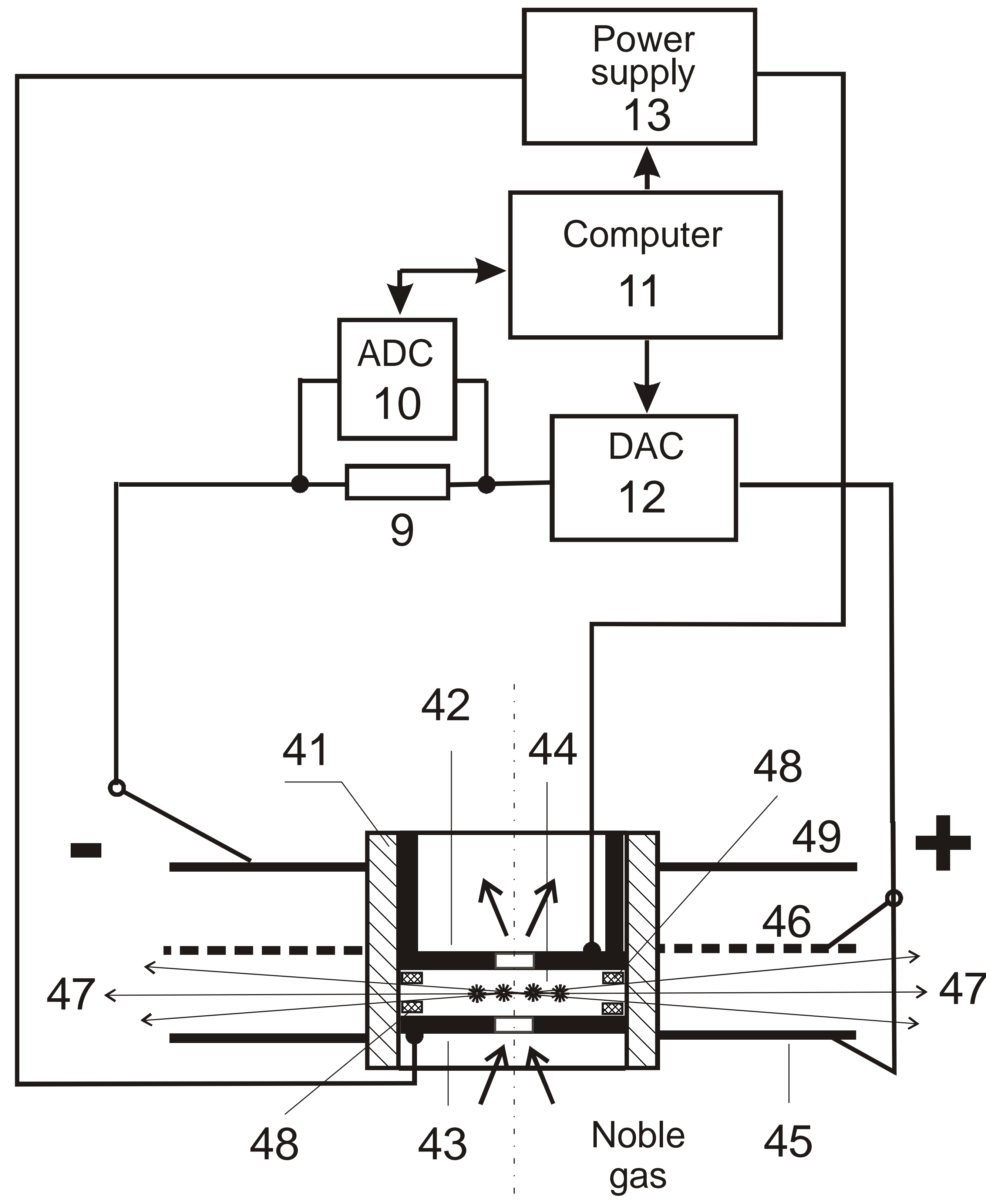| Chemical formula	Name	Ionization potential	
C_3H_8O	Ethyl methyl ether	9.72	0.07
C_4H_{10}	Butane	10.53	0.8
$C_4H_{10}O$	2-Butanol	9.88	0.02
$C_4H_{10}O$	2-Methyl-1-propanol	10.02	0.04
$C_4H_{10}O$	2-Methyl-2-propanol	9.90	0.02
$C_4H_{10}O$	Diethyl ether	9.51	0.02
$C_4H_{10}O$	Methyl propyl ether	9.41	0.07
$C_4H_{10}O$	Isopropyl methyl ether	9.45	0.04
C_5H_{12}	Pentane	10.28	0.9
C_5H_{12}	Isopentane	10.32	0.04
C_5H_{12}	Neopentane	(10.2)	
$C_5H_{12}O$	1-Pentanol	(10.00)	
$C_5H_{12}O$	2-Pentanol	(9.78)	
$C_5H_{12}O$	3-Pentanol	(9.78)	
$C_5H_{12}O$	2-Methyl-1-butanol	(9.86)	
$C_5H_{12}O$	2-Methyl-2-butanol	(9.8)	
$C_5H_{12}O$	3-Methyl-2-butanol	(9.88	0.13)
$C_5H_{12}O$	Butyl methyl ether	(9.4	0.1)
$C_5H_{12}O$	Methyl tert-butyl ether	(9.24)	
$C_5H_{12}O$	Ethyl propyl ether	(9.45)	
$C_6H_{11}NO$	Caprolactam	(9.07	0.02)
C_6H_{14}	Hexane	(10.13)	
C_6H_{14}	2-Methylpentane	(10.12)	
C_6H_{14}	3-Methylpentane	(10.08)	
C_6H_{14}	2,2-Dimethylbutane	(10.06)	
C_6H_{14}	2,3-Dimethylbutane	(10.02)	
$C_6H_{14}O$	1-Hexanol	(9.89)	

Russian patent No. 2217739

РОССИЙСКАЯ ФЕДЕРАЦИЯ

ПАТЕНТ

НА ИЗОБРЕТЕНИЕ

№ 2217739

Российским агентством по патентам и товарным знакам на основании Патентного закона Российской Федерации, введенного в действие 14 октября 1992 года, выдан настоящий патент на изобретение

СПОСОБ АНАЛИЗА ГАЗОВ И ИОНИЗАЦИОННЫЙ ДЕТЕКТОР ДЛЯ ЕГО ОСУЩЕСТВЛЕНИЯ

Патентообладатель(ли):

Кудрявцев Анатолий Анатольевич, Цыганов Александр Борисович

по заявке № 2002128663, дата поступления: 18.10.2002

Приоритет от 18.10.2002

Автор(ы) изобретения:

Кудрявцев Анатолий Анатольевич, Цыганов Александр Борисович

Патент действует на всей территории Российской Федерации в течение 20 лет с **18 октября 2002 г.** при условии своевременной уплаты пошлины за поддержание патента в силе

Зарегистрирован в Государственном реестре изобретений Российской Федерации

г. Москва, **27 ноября 2003 г.**

Генеральный директор
А.Д. Корчагин

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(51) 7 G 01 N 27/62

РОССИЙСКОЕ АГЕНТСТВО
ПО ПАТЕНТАМ И ТОВАРНЫМ ЗНАКАМ

(12) **ОПИСАНИЕ ИЗОБРЕТЕНИЯ**
к патенту Российской Федерации

(54) СПОСОБ АНАЛИЗА ГАЗОВ И ИОНИЗАЦИОННЫЙ ДЕТЕКТОР ДЛЯ ЕГО ОСУЩЕСТВЛЕНИЯ

(57) Изобретения относятся к средствам определения состава газовых смесей и позволяют производить качественный и количественный анализ примесей в основном газе. Сущность изобретения: определение примесей осуществляется путем анализа энергии электронов, образующихся при ионизации атомов или молекул примесей при столкновениях с частицами определенной

Блок питания 13

Компьютер 11

АЦП 10

ЦАП 12

Ключ 14

Источник 15

Фиг. 1

US patent 7,309,992

The Director of the United States Patent and Trademark Office

Has received an application for a patent for a new and useful invention. The title and description of the invention are enclosed. The requirements of law have been complied with, and it has been determined that a patent on the invention shall be granted under the law.

Therefore, this

United States Patent

Grants to the person(s) having title to this patent the right to exclude others from making, using, offering for sale, or selling the invention throughout the United States of America or importing the invention into the United States of America for the term set forth below, subject to the payment of maintenance fees as provided by law.

If this application was filed prior to June 8, 1995, the term of this patent is the longer of seventeen years from the date of grant of this patent or twenty years from the earliest effective U.S. filing date of the application, subject to any statutory extension.

If this application was filed on or after June 8, 1995, the term of this patent is twenty years from the U.S. filing date, subject to any statutory extension. If the application contains a specific reference to an earlier filed application or applications under 35 U.S.C. 120, 121 or 365(c), the term of the patent is twenty years from the date on which the earliest application was filed, subject to any statutory extensions.

Jon W. Dudas

Director of the United States Patent and Trademark Office

US007309992B2

(12) **United States Patent**
Kudryavtsev et al.

(10) Patent No.: **US 7,309,992 B2**
(45) Date of Patent: **Dec. 18, 2007**

(54) **GAS ANALYSIS METHOD AND IONISATION DETECTOR FOR CARRYING OUT SAID METHOD**

(76) Inventors: **Anatoly Anatolievich Kudryavtsev**, ul. Kropotkina, d. 15, kv. 47, 197101 St.-Petersburg (RU); **Alexander Borisovich Tsyganov**, ul. Nalichnaya, d. 45, korpus 1, kv. 5, 199397 St.-Petersburg (RU)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 228 days.

(21) Appl. No.: **10/531,536**

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(86) PCT No.: **PCT/RU03/00454**
§ 371 (c)(1),
(2), (4) Date: **Apr. 15, 2005**

(87) PCT Pub. No.: **WO2004/036206**
PCT Pub. Date: **Apr. 29, 2004**

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(51) Int. Cl.
G01N 27/62 (2006.01)
(52) U.S. Cl. **324/464**; 324/459; 250/282
(58) Field of Classification Search 324/464, 324/459; 250/282
See application file for complete search history.

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Primary Examiner—Vincent Q. Nguyen
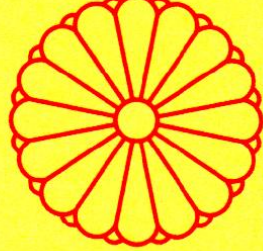(74) *Attorney, Agent, or Firm*—J. Herbert O'Toole; Nexsen Pruet, LLC

(57) **ABSTRACT**

Chemical analysis of impurities in buffer gas is provided at various pressures up to atmospheric. Identification of the impurities is carried out by analyzing energy of electrons releases via ionization of the impurity atoms or molecules in their collisions either with excited atoms of buffer gas or with monochromatic photons. To produce excited metastable atoms a pulsed plasma is ignited between plane anode and cathode, and electrons energy is measured in afterglow by determining second derivative of electric current in dependence of voltage applied between these electrodes. Another way, electrons energy can be analyzed by positioning a grid between anode and cathode and by using an external radiation source to ionize the impurities in equipotential space between the grid and the anode. Inter-electrode gap and gas pressure must be chosen so that distortions in the electrons energy distribution due to collisions with buffer gas should not exceed a prescribed value.

41 Claims, 9 Drawing Sheets
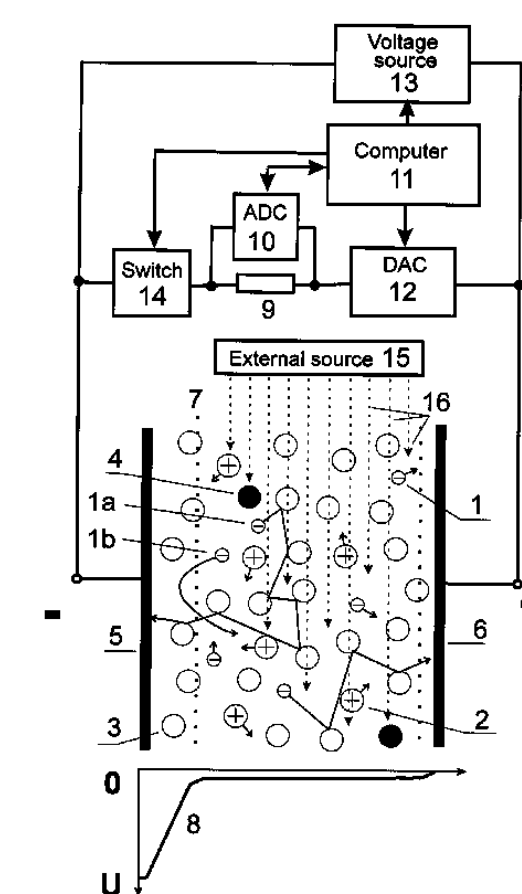
Japan patent No. 4408810

特許証
(CERTIFICATE OF PATENT)

特許第４４０８８１０号
(PATENT NUMBER)

発明の名称(TITLE OF THE INVENTION)

ガス分析方法とイオン化検出器

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出願番号(APPLICATION NUMBER)　　　特願２００４‐５４５１１３

出願年月日(FILING DATE)　　　　　平成１５年１０月１５日(October 15.2003)

この発明は、特許するものと確定し、特許原簿に登録されたことを証する。
(THIS IS TO CERTIFY THAT THE PATENT IS REGISTERED ON THE REGISTER OF THE JAPAN PATENT OFFICE.)

平成２１年１１月２０日(November 20.2009)

特許庁長官(COMMISSIONER, JAPAN PATENT OFFICE)

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氏　名
長谷川　芳樹　　　　　　　様

特許権設定登録通知書

特許番号	第４４０８８１０号
登録日	平成２１年１１月２０日
出願番号	特願２００４‐５４５１１３
出願日	平成１５年１０月１５日
請求項の数	４１
納付年分	第　３年分まで
受領金額	３１，５００円
受領日	平成２１年１１月１０日

特許料の納付について

・特許権を維持するには、存続期間の満了（特許出願の日から２０年）までの各年について所定の特許料の納付が必要です。

・第４年以降の各年分の特許料は、登録日（出願公告を経て特許になった場合は、公告日）の翌日を起算日として、納付済年分の満了日（以下「納付期限日」という）までに、次の年分の納付が必要です。

・納付期限日までに納付できなかったときは、その期間の経過後６ヶ月以内であれば特許料を追納することができます。

・追納する場合は、納付すべき特許料のほか、その特許料と同額の割増特許料が必要です。

・追納できる期間内に納付しないときは、その特許権は、納付期限日にさかのぼって消滅したものとみなされます。
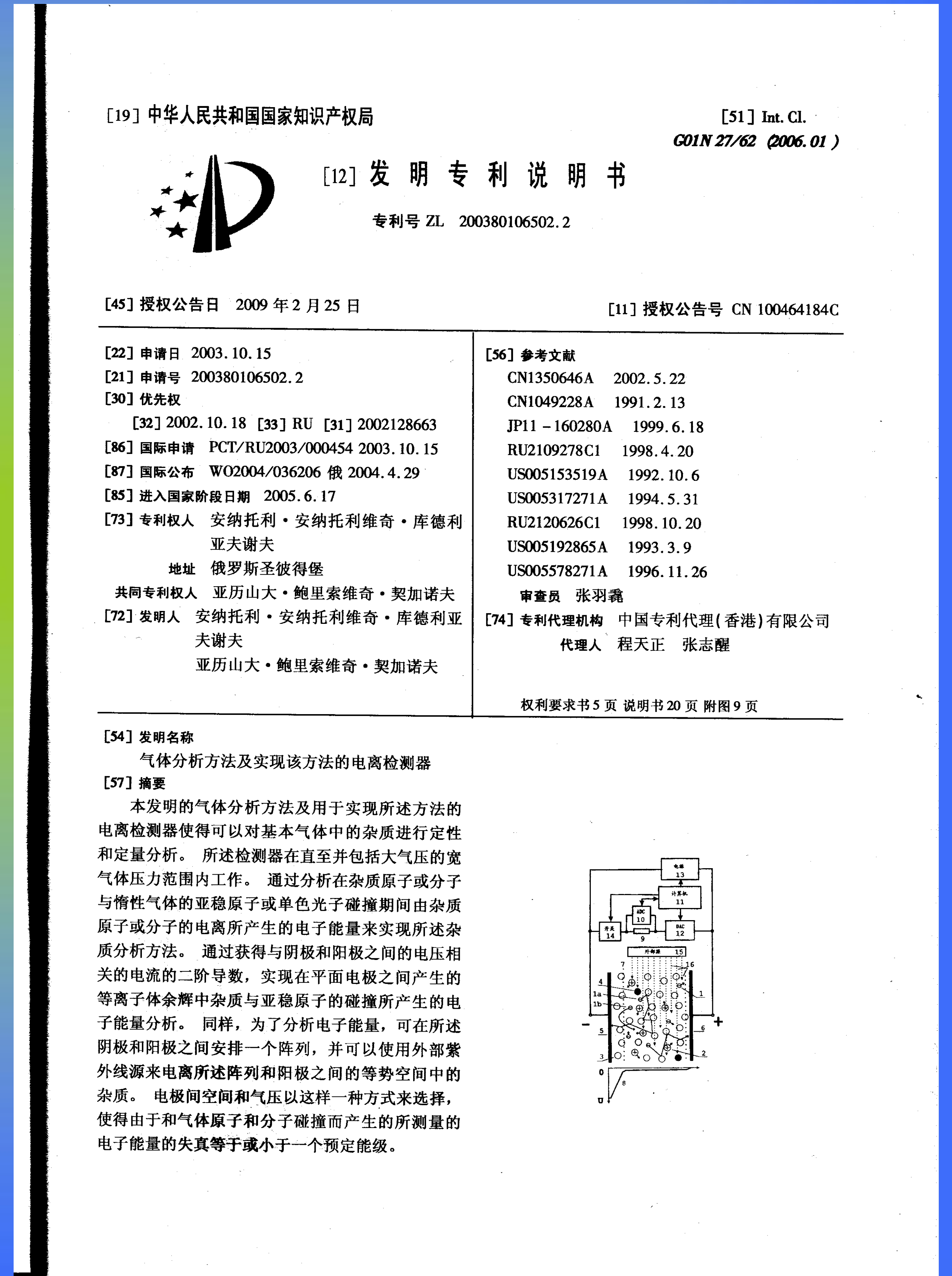
・特許料納付書の様式及び特許料の額については、以下を参照してください。

特許庁ホームページ
http://www.jpo.go.jp/indexj.htm

特許料納付期限日

納付年分	納付期限日
第　４年分	平成２４年１１月２０日
第　５年分	平成２５年１１月２０日
第　６年分	平成２６年１１月２０日
第　７年分	平成２７年１１月２０日
第　８年分	平成２８年１１月２０日
第　９年分	平成２９年１１月２０日
第１０年分	平成３０年１１月２０日
第１１年分	平成３１年１１月２０日
第１２年分	平成３２年１１月２０日
第１３年分	平成３３年１１月２０日
第１４年分	平成３４年１１月２０日

（注）納付期限日が行政機関の休日にあたるときは、その日の翌日が期間の末日となります。

問い合わせ先　出願支援課登録室
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China patent　ZL200380106502.2

左：发明专利证书

证书号 第472701号

发明专利证书

发 明 名 称：气体分析方法及实现该方法的电离检测器

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授权公告日：2009 年 2 月 25 日

本发明经过本局依照中华人民共和国专利法进行审查，决定授予专利权，颁发本证书并在专利登记簿上予以登记。专利权自授权公告之日起生效。

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局长　田力普

2009 年 2 月 25 日

第 1 页（共 1 页）

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权利要求书 5 页 说明书 20 页 附图 9 页

[54] 发明名称
气体分析方法及实现该方法的电离检测器

[57] 摘要

本发明的气体分析方法及用于实现所述方法的电离检测器使得可以对基本气体中的杂质进行定性和定量分析。所述检测器在直至并包括大气压的宽气体压力范围内工作。通过分析在杂质原子或分子与惰性气体的亚稳态原子或单色光子碰撞期间由杂质原子或分子的电离所产生的电子能量来实现所述杂质分析方法。通过获得与阴极和阳极之间的电压相关的电流的二阶导数，实现在平面电极之间产生的等离子体余辉中杂质与亚稳态原子的碰撞所产生的电子能量分析。同样，为了分析电子能量，可在所述阴极和阳极之间安排一个阵列，并可以使用外部紫外线源来电离所述阵列和阳极之间的等势空间中的杂质。电极间空间和气压以这样一种方式来选择，使得由于和气体原子和分子碰撞而产生的所测量的电子能量的失真等于或小于一个预定能级。

European patent EP1557667 (Germany, France, UK)

(54) **GASANALYSEVERFAHREN UND IONISATIONSDETEKTORZUR AUSF HRUNG DES VERFAHRENS**

(57) Ein Verfahren zur Gasanalyse und ein Ionisationsdetektor zur Durchführung des Verfahrens erlauben, eine qualitative und quantitative Analyse von Verunreinigungen in einem Grundgas vorzunehmen. Der Detektor kann in einem breiten Gasdruckbereich sogar bis zum atmosphärischen Druck arbeiten. Die Bestimmung der Verunreinigungen erfolgt durch die Analyse der Energie von Elektronen, die sich bei der Ionisierung der Atome oder Moleküle der Verunreinigungen bei den Zusammenstössen mit metastabilen Atomen eines Edelgases oder mit monochromatischen Photonen bilden. Die Analyse der Energie von Elektronen, die sich bei den Zusammenstössen der Verunreinigungen mit den metastabilen Atomen in einem Nachleuchtplasma bilden, das zwischen flachen Elektroden gezündet wird, wird durch das Erzeugen der zweiten Ableitung des Stroms in Abhängigkeit von der Spannung zwischen der Anode und der Kathode vorgenommen. Für die Analyse der Energie von Elektronen kann auch ein Gitter zwischen der Anode und Kathode angebracht werden und zur Ionisierung der Verunreinigungen in einem Äquipotentialraum zwischen dem Gitter und der Anode eine äußere UV-Strahlungsquelle einsetzen. Der Abstand zwischen den Elektroden und der Gasdruck werden so gewählt, dass Verfälschungen der gemessenen Energie von Elektronen, die durch die Zusammenstöße mit den Atomen oder Molekülen des Gases bedingt sind, ein vorgegebenes Niveau nicht überschreiten.

Fig.1
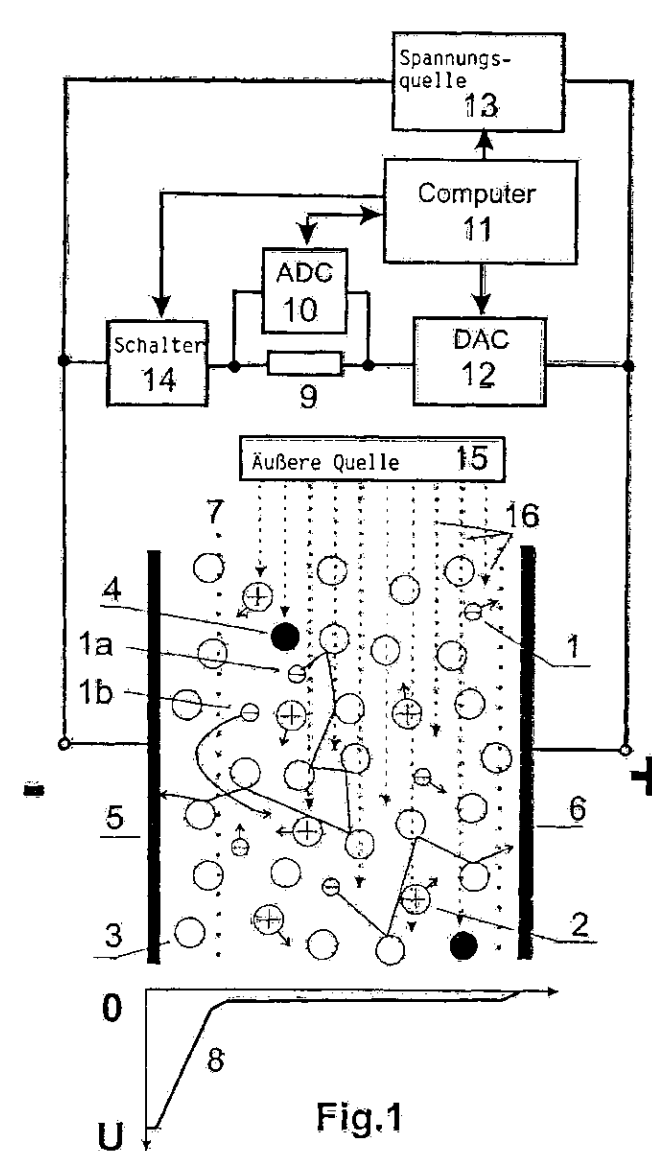
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