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(54) Benevnelse **SAMPLE RECEIVING ELEMENT, ANALYSES SET AND METHOD FOR ANALYZING A LIQUID, IN PARTICULAR A COOLING LUBRICANT EMULSION**

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Description

The invention relates to a sample-holding element for a liquid sample, to an analysis device set for simultaneous analysis of three or more physico-chemical parameters of liquids, which comprises an analysis apparatus designed as a hand-held device and the sample-holding element for the liquid sample, to the use of the set and to a method, in order to carry out which the analysis apparatus is used while employing the sample-holding element.

From the prior art, measuring apparatuses are known with which various parameters of cooling lubricants may be measured or checked. For instance, refractometers, with which the refractive index of the cooling lubricant may be determined. In the case of water-mixed cooling lubricants, the mixing ratio may be deduced from the refractive index. Measuring apparatuses for determining electrical conductivity are furthermore known, in which the resistance over a particular measurement section is determined. There are furthermore measuring apparatuses for determining the pH of cooling lubricants. Essentially, two types of pH measuring apparatuses are in this case used: pH metres with an electrode and optochemical pH measuring apparatuses.

There are furthermore measuring apparatuses which can determine different parameters after prior selection of the measurement quantity.

For instance, DE 10 2010 028 319 discloses a method for controlling the concentration of water-mixed cooling lubricants, in which a refractometer is used in order to determine the refractive index of the cooling lubricant and the electrical conductance is recorded by a resistance measurement, the inverse of which gives the conductance. The temperature of the cooling lubricant is furthermore monitored in order to take into account the changes in the data resulting from temperature variations. Conclusions about the composition of the cooling lubricant are drawn from the measured quantities, and this composition is adapted if required.

DE 696 34 490 T2 discloses a disc-shaped microsystem platform with two flat planar surfaces as a sample-holding element of a liquid sample. The disc-shaped microsystem platform comprises an input signal for a liquid sample, liquid microchannels, reaction and detection chambers, a plurality of measuring points of the analysis of the liquid sample being provided on the disc. The measurements which may be carried out comprise luminescence measurements and refractive index measurements, as well as electrochemical detection methods. The associated analysis device is similar to a CD player equipped with elements for rotating and reading the disc-shaped microsystem platform in order to control the functions. After application of the analyte to be tested into the input store, the microsystem platform is inserted into the CD player device, the liquid transport through the microchannels is on the disc taking place by means of centripetal acceleration in the CD player device and by selective activation of valves on the disc. The analysis results may be stored and/or displayed immediately to the user.

A sample cartridge with channels and chambers, which may comprise electrodes and detection windows for optical measurements, is known from US 2011/201099 A1. The chambers may contain reagents such as binding reagents, detectable markings, sample preparation reagents, wash solutions, buffer, etc., in liquid or solid form or on the surface of solid immobilized phase carriers. The associated analysis apparatus, which is not designed as a hand-held device, comprises corresponding detectors for the measurements to be carried out and means for receiving the cartridge and for positioning the cartridge, as well as electrical systems for contacting the electrodes of the cartridge and control systems for recording, processing and storing the signals of the detectors. For luminescence measurements, the analysis apparatus comprises an area closed in a light-tight manner. In order to receive and position the sample-holding element, the analysis apparatus comprises a cartridge tray, which is mounted on rails by means of a guide carriage in order to permit motor-driven movement of the tray into and out of the area which is closed in a light-tight manner.

US 2013/330245 A1 describes a sample-holding element with channels and a detection chamber for optical measurements, as well as electrodes at the fluid inlet

for measuring the resistance of the sample in order, during the sampling, to signal sufficient immersion of the sample-holding element into the liquid to be tested. For the sampling, the sample-holding element is introduced into the associated analysis apparatus designed as a hand-held device, which can receive a plurality of sample-holding elements simultaneously for analysis of a plurality of parameters and comprises corresponding optical analysis device, etc. During the sampling, a liquid sample is drawn into the sample-holding element by a pump of the analysis device.

On the basis of this prior art, it is an object of the present invention to provide an improved device with which a liquid - such as a cooling lubricant - can be prepared by very simple handling, with a minimized sample quantity outlay, for measurement of the concentration of the liquid or of its components and for measurement or determination of a plurality of further parameters such as refractive index and temperature, and optionally further parameters which characterise the constitution of the fluid.

This object is achieved with the aid of a sample-holding element having the features of Claim 1.

The further object, to provide an improved device with which the concentration of a liquid such as a cooling lubricant, together with further parameters such as refractive index and temperature, and optionally yet further parameters which characterise the constitution of the fluid, can be reproducibly and reliably measured or determined in an improved way directly in situ, is achieved by the analysis device set having the features of Claim 7.

The yet further object of providing an improved and at the same time simplified measurement method for analysis of more than three parameters of a liquid such as a cooling lubricant in situ is achieved by the method having the features of independent Claim 15.

Refinements and preferred embodiments of the devices and of the method are mentioned in the dependent claims.

A first embodiment of the sample-holding element according to the invention for a liquid sample, for example a cooling lubricant, for simultaneous analysis of three or more chemico-physical parameters of the liquid, i.e. essentially characteristic data thereof, by means of an analysis device, primarily in order to determine the concentration of at least one constituent thereof, comprises, besides a sample-holding space that can be filled with the liquid, at least three measuring points in adjacent arrangement with respect to each other, at least one of which is a photonic measuring point (this includes measuring points for absorption and photoluminescence) and another is a refractive index measuring point.

The sample-holding element furthermore comprises at least one further measuring point, which may for example be a pH measuring point, a conductivity measuring point or a germ measuring point. All the measuring points are distributed across the sample-holding space, i.e. particular areas of the sample-holding space respectively form a measuring point, so that the measuring points are in fluidic contact with the liquid when liquid is received in the sample-holding space of the sample-holding element.

A “measuring point” therefore refers to a predetermined area of the sample-holding space, which is correspondingly designed for the measurement respectively intended there on the liquid: If intended measurement requires an optoelectronic measuring device, for instance for recording photoluminescence, with which light is directed onto the predetermined area of the sample-holding space and the luminescence emitted by the liquid is recorded, the measuring point of the sample-holding element correspondingly comprises transparent windows on both sides of the sample-holding space in the corresponding area, on which (the windows) the liquid bears directly. In another example, if the measurement to be carried out is intended to determine the conductivity by means of the electrical resistance of the liquid, the electrodes extend into the sample-holding space, the measuring point being formed by the distance (“measurement section”) between the electrodes, which is in direct contact with the liquid when sample-holding space has been filled.

For the adjacent arrangement of a plurality of measuring points, the sample-holding element according to the invention is a flat element that is configured to be double-walled and has plates arranged in a plane-parallel fashion on one another and connected to one another at least in sections at their edges. For the refractive index measuring point, one of the plates in this case comprises a prism structure in the area predetermined for this purpose, preferably on the inner side of the plate, with which prism structure light rays which penetrate through the other plate and pass through the sample-holding space (and the liquid contained therein) are refracted in a predetermined manner, the plates being transparent in the predetermined area for the wavelengths used for the refractive index measurement. In this case, the sample-holding space is designed in the form of a gap in planar manner between the two plates, the distance between the double walls being just so large that a liquid sample can be drawn into the sample-holding space by means of the capillary effect at least in a position at which the plates are not connected to one another at the edge. In the case of aqueous emulsions with a water content of at least 20%, this distance may lie in the range of from 0.5 to 2 mm, preferably about 1 mm. If a liquid to be analysed has a water content differing therefrom or a different viscosity, a sample-holding element is configured with a correspondingly adapted distance between the plates, in order as before to achieve filling of the sample-holding space caused purely by capillary forces. Thus, it is sufficient to immerse the sample-holding element with the opening, which is formed by the unconnected edge positions, into the liquid which is intended to be studied, or to touch the liquid surface - since complete immersion is not necessary - whereupon the liquid sample flows into the sample-holding space by the capillary effect. Precisely for fluids such as cooling lubricants, this is a very suitable method. Advantageously, with the purely passive filling by the capillary effect, no auxiliary means are required in order to bring the liquid sample into the sample-holding space of the sample-holding element, as is the case in the prior art, in which either pipettes are used for the sampling and filling of the sample-holding element, which then carries out a rotational movement to bring the liquid to the measuring points, or sample-holding elements are used which need to be connected to a pump in order to draw the liquid into the sample-holding space and to the measuring points.

Thus, advantageously, an extremely small sample volume is sufficient in order to measure a multiplicity of three, or rather four or more, different chemico-physical parameters.

The sample-holding element designed as a planar element may suitably be a planar elongate sample strip with an overall thickness which lies in the range of from 2 to 8 mm, preferably in the range of from 2.5 to 6 mm, and particularly preferably in the range of from 2.5 to 4.5 mm.

In this case, for the design of a sample-holding element that is as small as possible, it is necessary to ensure that the measuring points lie as close to one another as possible, essentially on a fluid path that leads from the inlet opening, which may be designed in the form of a gap - there may also be a plurality of inlet openings – along the measuring points along an outlet position, provided as a venting channel with an air exit opening, at which the plates are likewise not connected to one another.

In order to allow convenient filling, the plates are preferably not connected to each other at least along one side of the planar sample-holding element, in particular designed as an elongate sample strip, so that a filling gap for the liquid is provided. This may preferably be a long side of the sample strip, since a much shorter filling time of the sample-holding space may be achieved by the longer filling gap than by a filling gap on a short side.

Thus, a narrow thin test strip consisting of two platelets may be provided, on one along side of which the platelets are not connected, or adhesively bonded, to one another at the edge at one or more positions, and in the interior of which, because the platelets are also not adhesively bonded there, a thin gap like a channel is formed, which guides the liquid and along which the measuring points lie. For complete filling of the sample-holding space, the venting channel may extend through a short side and open outwards in order to release air which is displaced from the sample-holding space during the sampling by ingress of the liquid.

Since the sample-holding element comprises measuring points at which optical or optoelectronic analysis methods may apply, the planar element is advantageously at least in part - i.e. at least in the area of the measuring points intended therefor - made from an optically transmissive material such as glass, or quartz glass, or from a transparent plastic, for example polymethyl methacrylate or polycarbonate. Other transparent plastics may, however, also be envisaged.

In order to form the measuring point for the conductivity measurement, at least two contact strips of the application of voltage are arranged on an extended section of one of the plates, which protrudes beyond the other plate, which extend as electrodes into the sample-holding space and end there separated by a measurement section that forms the conductivity measuring point.

Advantageously, according to another embodiment, the planar element is designed, at an end that faces away from the end with the contact strips of the conductivity measuring point, in the form of a handle section for handling of the sample-holding element. The venting channel starting from the sample-holding space may extend through this sample section and emerge there at an air exit opening. Since, in combination with the analysis apparatus according to the invention, designed as a hand-held device, it is provided that the handle section of the sample-holding element protrudes partially from the analysis apparatus during the measurement process, a different arrangement of a venting channel may also be provided, for instance when it is intended as a measuring point for a germ measurement by means of gas (micro)sensors. A venting channel starting from the sample-holding space then emerges at a different position, where its air or gas exit opening may communicate with corresponding gas (micro)sensors of an analysis apparatus.

The handle section may furthermore be opaque, preferably black, in order to prevent stray light incidence when the sample-holding element has been introduced into the analysis apparatus. It is, however, also conceivable to provide differently coloured handle sections for different sample-holding elements. A sample-holding element which is received fully by an analysis apparatus may also be made entirely transparent. Furthermore, a handle section may comprise a structured surface in

order to facilitate handling by better gripping. Furthermore, markings may be applied on the handle section - or on other suitable positions of the sample-holding element - in order to assist correct introduction of the sample-holding element into an analysis apparatus. Correct insertion may be assisted according to the key-lock principle or by special shaping of the sample-holding element, in particular on the end facing away from the handle section.

Preferably, the photonic measuring point is a luminescence measuring point, and particularly preferably a fluorescence measuring point. To this end, the sample-holding element comprises a window section, which is transparent to the corresponding excitation and emission wavelengths, on both plates in the area intended for the measuring point. The two window sections of the measuring point for the luminescence measurement may be congruent.

If the sample-holding element comprises a pH measuring point, this may comprise an indicator dye-containing substrate that is arranged at a predetermined second section between the two plates, which are correspondingly transparent to the light required for the optoelectronic recording of the colour change of the indicator substrate in an area that encloses this section.

Furthermore, the group from which the at least one further measuring point is selected may comprise a nitrite measuring point, which may be formed in a manner comparable to the pH measuring point in relation to optoelectronic recording of a colour change, but with a nitrite reactive substrate. To this end, for example, a primary aromatic amine may be envisaged, which reacts with nitric acid to form a diazonium salt that in turn forms, in acidic solution with amines, a coloured azo compound which is photometrically detectable and quantifiable with calibration. One known reagent (Griess' reagent, photometric detection of the azo compound at 535 nm) consists of 1-naphthylethylenediamine and sulfanilic acid and optionally acetic acid. Analytical monitoring of the nitrite content in aqueous metal-processing solutions or emulsions is important since nitrite can react as a reaction partner with secondary amines or alkanolamine to form carcinogenic nitrosamines. Nitrite may inter alia by means of the preparation water lead to

emulsification or enter the process media, i.e. the aqueous metal-processing solutions or emulsions, by means of the metal parts which undergo a hardening process and are still contaminated with hardening salts.

In order to form the refractive index measuring point, according to the invention the prism structure or a Fresnel lens structure, which represents a special prism structure, provided at a predetermined third section of one of the two plates. In this case as well, the plates are transparent at the section to the wavelengths used for the refractive index measurement. The prism structure provides surface sections that are angled with respect to the plate plane, at which incident light rays are refracted accordingly. A prism structure consists of at least one, preferably a plurality of, structures with a triangular profile in an adjacent arrangement. A Fresnel lens structure comprises a series of ring-shaped steps.

The section with the prism structure, or the Fresnel lens structure, and the section with the indicator dye-containing substrate in this case form constituent components for the optical, electronic and optoelectronic analysis apparatuses, with which the sample-holding element corresponds during a measurement, or an analysis process.

In general, the sample-holding element is in this case configured as a single-use measurement strip.

An analysis device set, likewise according to the invention, for simultaneous analysis of three or more chemico-physical parameters, or characteristic data of liquids comprises an analysis apparatus designed as a hand-held device with a housing and with a display, as well as at least one sample-holding element according to the invention for the liquid sample. In this case, a hand-held device means that the device is small and handleable and may easily be carried by a person to the systems which use the liquid to be analysed, and can be manually operated. For the measurements to be carried out on the sample-holding element, the analysis apparatus comprises an optoelectronic analysis device which comprises at least three measuring devices in an adjacent arrangement with respect to each other,

whose arrangement matches the arrangement of the measuring points on the sample-holding element. The analysis apparatus furthermore comprises a data processing unit that is connected in communicative manner to the analysis device and the display device.

In the housing of the analysis apparatus according to the invention, there is an insertion device for accommodation of the sample-holding element that is arranged in the housing in detachable manner and comprises an insertion opening. The latter terminates in a recess that is designed to match the sample-holding element for accommodation thereof. The insertion device furthermore comprises an optical, electronic or optoelectronic communication facility that matches the arrangements of the measuring devices and measuring points depending on the type of the respective measuring point, which is equipped with corresponding signal transmission (this is also intended to mean light transmission) between the measuring points of a sample-holding element accommodated in the insertion device and the measuring devices.

To this end, the insertion device is made at least partially from transparent material. This means that it is transparent at least at the positions where this is required for the optical measurements. In general, the insertion device may be made from opaque material, preferably from plastic, particularly preferably from black plastic, and is then also insensitive to stray light.

The insertion device may be designed with a flange section, which comprises the insertion opening, and with a shell section that is arranged in the housing such as to be detachable, borders the recess and comprises the optical, electronic or optoelectronic communication facilities. Although these are preferably formed in the manner of windows by sections made of transparent material - since only in this way is contamination of the interior of the analysis apparatus prevented - it is however also conceivable for these communication facilities merely to be formed by openings in the shell section. By the optical, electronic or optoelectronic communication facilities, the components of the analysis apparatus and of the sample-holding element can interact in order to make the analysis of the chemico-

physical parameters to be determined possible. In relation to the refractive index measurement, for example, the section with the prism structure and a corresponding light source are in communication through a window in the insertion device, in such a way that light passes through the window and the liquid accommodated in the sample-holding space onto the section with the prism structure, and is refracted there. A further window on the other side of the insertion device allows communication with a sensor of the analysis apparatus in order to determine the angle of refraction.

Two of the measuring devices of the analysis apparatus are a photonic measuring device, preferably a luminescence measuring device, particularly preferably a fluorescence measuring device, which, when fluorescence markers are used in the liquid, is used for the concentration measurement, of one or a plurality of possibly different components of the liquid, and a refractive index measuring device. The luminescence measuring device comprises an excitation light source with a wavelength suitable for excitation of the fluorescence markers, and sensors suitable for measuring the emitted fluorescence. The refractive index measuring device of the analysis apparatus comprises, except for the prism structure which as mentioned above is part of the sample-holding element, all other required components of the refractometer, such as the light source and sensors.

In a similar way at least one further measuring point of the sample-holding element, the analysis apparatus comprises at least one further measuring device which is selected from the group in accordance with the measuring points of the sample-holding element. This may, for example, be a pH measuring device, which may preferably be configured as a pH optode, the optical effect of the colour change of the indicator substrate on contact with the liquid to be tested being used. If an indicator paper is used as the indicator substrate, a measuring device in which the light colour reflected by the indicator paper is detected is used.

Preferably, a universal indicator with a mixture of a plurality of indicator substances is used, with different colours and different transition regions which are

adapted so that pH values in a wide pH range can be made detectable by different colour transitions.

If the sample-holding element comprises a nitrite measuring point for detecting nitrite, or for measuring the nitrite content, an analysis apparatus correspondingly equipped with a nitrite measuring device is to be used for the analysis.

Thus, if a light source unit that comprises not only the light source but also possibly required optical components such as filters, lenses, etc., is respectively intended to be used, and a detection unit (likewise optionally comprising components such as filters, lenses, etc., and the actual detector) are provided both for the luminescence measuring device and for the refractive index measuring device, and also for the pH measuring device and the nitrite measuring device. The various measuring devices may comprise different light sources and detectors, which may be selected according to the measurement principle - this selection is known to the person skilled in the art. The light source units of the various measuring devices may be arranged in the analysis apparatus on one side of the sample-holding element, or the insertion device, and the detector units may be arranged on the other side. The beam paths between the light sources and detectors extend through a corresponding arrangement or the use of corresponding optical components so that the light rays pass through (luminescence and refraction) the sample-holding element at the respective measuring point or are reflected there (pH).

As a measuring device as an alternative or in addition to the pH measuring device, an analysis apparatus may also comprise a conductivity measuring device, which is in fact a resistance measuring device and with which the conductivity of the liquid is determined from the measured resistance. In this case as well, the sample-holding element with the contact strips comprises a part of the measuring device. The conductivity measuring device of the analysis apparatus comprises a frequency generator with contact elements which, depending on the arrangement of the sample-holding element in the analysis apparatus, are in electrical contact with the at least two contact strips of the sample-holding element directly or indirectly via contact bridge elements.

In order to record the germ load of the liquid, the analysis apparatus may comprise a corresponding measuring device, which may be a so-called “electronic nose” that is formed at least from one microelectronic gas sensor, usually from a multiplicity of gas sensors, since germs produce volatile organic compounds that pass from the liquid into the vapour phase and can be detected with the gas sensors when this vapour phase is brought into communication with the sensors. To this end, the venting channel of the sample-holding element may be brought into communication with the electronic nose via a connecting line of the analysis apparatus. The connecting line may also lead to the filling gap - conceivably, as a germ measuring point, there could also be a correspondingly gas-permeable window in at least one of the plates, via which the volatile compounds may pass through the connecting line to the gas sensors. In order to obtain a directed feed flow of the volatile compounds to the gas sensors, the use of a microblower is conceivable; directed flow guidance may also be assisted by a special design of the venting channel and the connecting line in terms of cross-sectional configuration.

Since, in particular, the refractive index is temperature-dependent, the analysis apparatus comprises a temperature measuring device, which is connected to the data processing unit so that the influence of the temperature during the measurement of the refractive index can be compensated for. The temperature sensor used may for example be a resistance thermometer, which because of its small size can be accommodated well in the housing of the analysis apparatus designed as a hand-held device.

Contamination of the sensitive measurement technology inside the analysis apparatus is prevented by the already described insertion device, which separates the inserted sample-holding element from the interior of the analysis apparatus, the housing of which is designed to be correspondingly fluid- and dust-tight. The flange section of the insertion device of a preferred embodiment, in an analytical arrangement in which the insertion device is inserted into the housing, touches, on the outside, against an edge of the housing and frames a cover plate, into which the insertion opening is introduced. This insertion opening may be sealed by a sealing

lip, or a pair of sealing lips, so that liquid possibly present on the outer side of the sample-holding element can be wiped off during insertion and therefore does not enter the analysis apparatus. The sealing lip(s) are held in the flange section by the cover plate, the cover plate being fastened, for example screwed, in the flange section such as to be detachable. In this case, it is also possible that the screws are not only designed for fastening the cover plate in the flange section, but to pass through the flange section and therefore at the same time and ensure detachable fastening of the insertion device on the housing of the analysis apparatus. Other variants of the fastening both of the cover plate in the flange section and of the insertion device in the analysis apparatus are, however, also conceivable; thus, plug-in, clamping or latching systems may also be envisaged.

For the conductivity measurement, as an alternative to the above-described direct contacting of the contact strips of the sample-holding element with the contact elements of the frequency generator, the insertion device may comprise contact bridges that establish the contact of the contact elements of the analysis apparatus to the at least two contact strips of the sample-holding element, when the latter is arranged in the analytical arrangement in the insertion device.

The contact bridges and/or contact elements of the analysis apparatus may be designed as contact springs or a spring contact rail, in order to achieve secure contacting to the contact strips of the inserted sample-holding element.

The frequency generator and all other electrical consumers of the analysis apparatus, such as the optoelectronic analysis device, the data processing unit and the display device, as well as the thermal sensor, etc., are connected to an energy source that is likewise fitted in the housing of the analysis apparatus. The energy source may preferably be an accumulator, which can be recharged via an interface in the housing. Optionally, the analysis apparatus may also comprise one or more solar cells on the outer side for recharging the accumulator.

The display device may be designed as a touch-sensitive display device (also referred to below as a touchscreen display), and therefore at the same time represent

a control interface in order to transmit user inputs via the communication line to the data processing unit. The latter may comprise or be connected to an external communication interface, which may be a plug contact interface, for example a USB or micro-USB interface, or a radio interface, in particular a short-range radio interface, for example according to the Bluetooth® standard etc.

The invention also relates to a method for simultaneous analysis of at least three different chemico-physical parameters of a liquid in situ through the use of an analysis device set according to the invention. The method comprises the steps of:

- immersing the sample-holding element into the liquid or contacting an opening of the sample-holding element that is formed by the non-connected parts of the edge to the liquid surface, and filling the sample-holding space of the sample-holding element with a sample of the liquid to be tested through the action of the capillary effect between the double walls of the sample-holding element, to which end the filling opening is immersed in the liquid for a predetermined period of time that depends on the dimensions of the sample-holding space and of the filling opening,
- completely inserting the sample-holding element into the analysis apparatus,
- starting and carrying out at least three or more measuring processes simultaneously by means of the measuring devices at the measuring points,
- after completion of the measuring processes, displaying the measuring results on the display device.

Advantageously, in one refinement of the method, a liquid to be tested may be selected from various liquids that can be tested, which are deposited in a database, which is stored in the data processing unit or on a storage medium connected to it, and which are offered in a selection menu, through a user input on the display device, which may be designed in a suitable manner as a touchscreen display.

In general, however, it is possible to design the analysis apparatus for a particular type of liquid, in order to provide an apparatus that is particularly simple for a specific application, so that no liquid selection has to be carried out.

Likewise optionally, in refinements of the method, after completion of the measuring processes, a prompt for removal of the sample-holding element from the analysis apparatus may be displayed on the display device. The removal is detected by the software when the measuring process is completed. When the sample-holding element is inserted, however, the end position is detected optoelectronically and then the evaluation and data acquisition are started, which may be done automatically or by user input. Lastly, according to the invention it is also possible, after the removal of the sample-holding element from the analysis apparatus has been detected, to display the measuring results on the display device, and also to store the measuring results and/or transmit them to further apparatuses.

The storage may be carried out in an internal memory of the data processing unit or a removable storage medium connected thereto, for instance an SD card or a USB stick. The transmission of the measuring results may preferably be carried out via the radio interface to a preset receiver, but also in a wired manner by means of a corresponding USB cable.

Embodiments of the method relate to the calibration of the analysis apparatus for the liquids that can be tested, which are deposited in the database, and/or the training of new liquids with the analysis apparatus and addition of trained liquids to the database. Both, calibration and training, are respectively carried out by selection and confirmation of corresponding fields that are displayed in the selection menu, calibration solutions with known chemico-physical parameters being provided for the calibration in order to calibrate the measuring devices. In order to train new liquids, these are provided as liquids to be tested with known chemico-physical parameters.

In the method, the liquid is also a liquid that comprises at least one marker substance that can be detected by means of luminescence analysis, one of the measuring points being a luminescence measuring point.

In particular, the method may be applied while using an analysis device set according to the invention for analysing a metal processing liquid, in particular a

cooling lubricant, and in this case above all a cooling lubricant emulsion, as the liquid, at least one first marker substance that can be detected by means of luminescence analysis being added to the liquid at a predetermined concentration so that conclusions about the concentration of a liquid constituent, in particular the concentration of the cooling lubricant, in the emulsion are possible by means of the luminescence measurement.

In order to determine the cooling lubricant concentration in an emulsion by means of luminescence analysis, the marker substance is added to the cooling lubricant emulsion at a predetermined concentration. The molar concentration of the marker or of the marker composition, which may also be composed of a plurality of markers, is 10^{-5} to 10^{-6} mol/litre in the cooling lubricant concentrate, or 10^{-7} to 10^{-8} mol/litre in the application concentration, i.e. in the emulsion of the cooling lubricant. This dosage relates, inter alia to dyes with the basic chemistry of perylenes. The luminescence marker added to the liquid for the concentration measurement may be a dye that is invisible to the naked eye, or alternatively a visible dye.

Preferably, a marker may be used which is made up of at least two dye molecules from the range of rylene dyes, for example perylene or quaterrylene, or a combination of rhodamine carbonyl derivatives and acridine derivatives, so that at least two measurement ranges in the long-wave range can be covered. With simultaneous measurement in two measurement ranges, measurement errors can be minimized.

If the liquid is a cooling lubricant emulsion for special manufacturing purposes, a booster may be added for the purpose of increasing performance. This is usually done with a proportion of less than 5 percent by weight, expressed in terms of total weight of the cooling lubricant emulsion. For the manufacture of small batches of components with machine tools that are not actually intended for small batches, in order to maintain quality of the small batches such boosters need to be added in order to improve the performance of the cooling lubricant, in order to avoid a special cooling lubricant having to be developed for these requirements, which

would be uneconomical. In such cases, it is particularly advantageous to be able to determine the concentration of the booster with the aid of a further added marker, which labels the booster, straightforwardly in situ with the sample-holding element according to the invention and the associated analysis apparatus in the set. To date, it has only been possible to establish this in the laboratory by infrared spectroscopy in order to detect the ester band (when the booster contains an ester compound) and/or by X-ray fluorescence analysis in order to detect sulfur/phosphorus compounds of the booster.

Thus, the method according to the invention also relates to the liquid comprising a booster additive and at least one second marker substance that can be detected by means of luminescence analysis being added to the liquid at a predetermined concentration, the second marker substance differing from the first marker substance with regard to its luminescence properties, so that during the luminescence analysis, the concentration of a first constituent, for example the cooling lubricant in the emulsion can be deduced by means of the luminescence of the first marker, and the concentration of the booster additive can be deduced by means of the luminescence of the second marker.

The marker selection is dictated suitably according to viewpoints of the balance between oleophilicity and hydrophilicity. The marker, if only the booster is marked, also remains only in the booster during use and does not diffuse into the base emulsion. One explanation therefor could be the adjacent presence of different micellar structures. Thus, by means of particle measurement, for example by means of a Coulter counter, it is possible that such a so-called “two pack system” consisting of the booster and marker leads to two peaks, which implies the presence of different micellar structures. This theory could also be supported by such a two pack system generating a higher performance during use - in comparison with a system in which the key agent has been incorporated at a standard concentration. In this case, equal concentrations must also be considered for comparison.

Lastly, fluorescence measurements were also carried out on the marked emulsion systems.

The analysis device set according to the invention, consisting of a sample-holding element and an analysis apparatus, is therefore also very suitable for testing liquids such as metal processing liquids, cooling lubricants, cooling lubricant emulsions, which may also contain a booster.

Metal processing liquids are in the present case intended to mean all liquids which are used for lubrication and/or cooling, and possibly for washing, during metal processing operations such as shaping or machining processes such as cutting, grinding, lapping, insulation/erosion. In this case, cooling lubricants which combine the two functions of cooling and lubrication, and possibly also washing, are often used. Cooling lubricants with a minimal amount of lubrication may also be used. Cooling lubricant emulsions in turn relate to corresponding compositions mixed with water. Although the invention is particularly advantageously suitable for analysis of such metal processing liquids, or cooling lubricants, and in this case particularly aqueous cooling lubricant emulsions, it is in no way restricted thereto. Thus, a sample-holding element according to the invention, an analysis device set according to the invention and a method according to the invention may also be used in general for analysis of any fluids containing water, i.e. for example also gear or hydraulic fluids, or aqueous cleaning solutions or emulsions.

The liquid tested is preferably a liquid containing water, with a water content of from 1 to 99.9%, and it is particularly preferably a water-based liquid with a water content of from 1 to 15 %.

Further embodiments, and some of the advantages which are associated with these and further embodiments, will become clearly and better comprehensible through the following detailed description with reference to the appended figures. Objects, or parts thereof, which are substantially the same or similar may be provided with the same references. The figures are merely schematic representations of exemplary embodiment of the invention.

Fig. 1 shows a plan view of a sample-holding element according to the invention,

Fig. 2 shows a perspective view of an insertion device of an analysis apparatus according to the invention,

Fig. 2a shows a schematic sectional side view according to AA in Fig. 2,

Fig. 3 shows a side view of the insertion device,

Fig. 4 shows a side view of the analysis apparatus with an insertion device and an inserted sample-holding element,

Fig. 5 shows a schematic plan view of a half-shell of the analysis apparatus with an insertion device and an optoelectronic analysis device,

Fig. 6 shows a schematic representation of an optical pH measuring device of the optoelectronic analysis device,

Fig. 7 shows a schematic representation of a refractometer of the optoelectronic analysis device,

Fig. 8 shows a schematic representation of a luminometer of the optoelectronic analysis device,

Fig. 9 shows a schematic perspective view of an opened analysis apparatus with an insertion device,

Fig. 10 shows a side view of the analysis apparatus with an insertion device and an inserted sample-holding element of an alternative embodiment of the analysis device set,

Fig. 11 shows a plan view of a sample-holding element according to the invention with an additional nitrite measuring point,

Fig. 12 shows a schematic plan view of an optoelectronic analysis device of the analysis apparatus with an insertion device for the sample-holding element of the Fig. 11.

The analysis device set according to the invention relates to an analysis apparatus designed as a hand-held device for simultaneous determination of various characteristic data of a metal processing liquid, in particular a cooling lubricant, in a mobile manner in situ during manufacture or directly on the machine tool while using a special sample-holding element. Fig. 1 shows an exemplary sample-holding element 20, which is designed as a single-use test strip.

The sample-holding element 20 is in this case an approximately rectangular planar element, which comprises a sample-holding space 31 extended in a planar manner as a gap between two plates 30, 30', to which end the cover plate 30' is connected, except for an opening of length L provided for filling, at its edges to the base plate 30, which comprises various functional sections and elements. The filling opening may, as represented, be a continuous gap opening extending along a longitudinal edge; depending on the dimensions of the sample-holding element 20, however, a plurality of filling openings may also be provided, through which the sample-holding space 31 is filled by means of the capillary effect. Thus, the distance between the plates 30, 30' is selected to be just so large that the liquid sample can be drawn fully and uniformly into the sample-holding space 31 through the filling opening as a result of the capillary effect. The width of the gap is therefore also dependent on the dimensions of the sample-holding space 31, but lies in the range of from 0.1 to 2 mm, preferably 0.5 to 1.5 mm, for example about 1 mm, in order to form a sample-holding space 31. A suitable size for a sample-holding element 20 has, for example, been tested at 12 by 28 mm.

The sample-holding element 20 thus designed in Fig. 1 as a test strip may therefore have a thickness which lies in the range of from 2 to 8 mm, preferably in the range of from 2.5 to 6 mm, and particularly preferably in the range of from 2.5 to 4.5 mm. Furthermore, the size and shape of the sample-holding space 31, and therefore of the sample-holding element 20, also depends on the type, number and area

requirement of the measuring points 24, 25, 26, 27, all of which are adjacent inside the area of the sample-holding space 31 but mostly also need to be at a distance from one another.

The filling of the sample-holding space 31 is assisted by a venting channel 28, which extends between the plates 30, 30' to an air exit opening 29 - i.e. the plates are also not connected to one another in the area of the venting channel 28. In the example shown here, the venting channel 28 extends from a side of the sample-holding space 31 adjacent to the filling gap through a handle section 23. It is, however, also conceivable to vary the shape, number and arrangement of the venting channels.

The handle section 23 may be corrugated, or comprise other structures, for better handling.

Distributed across the sample-holding space 31, the sample-holding element 20 of Fig. 1 comprises three optical measuring points 24, 25, 26 in adjacent arrangement with respect to each other and a conductivity measuring point 27, which extend in the present example with two of their contact strips 22 into the region of one of the optical measuring points 24. With this sample-holding element 20, three optical measurements A, B, C and a conductivity measurement D can therefore be carried out simultaneously with the corresponding analysis apparatus 1 (cf. Fig. 9) after the liquid-filled sample-holding element 20 is being introduced through the insertion opening 9 into the analysis apparatus 1.

The first optical measuring point 24 is a photonic measuring point, which is in this case intended to mean all photonic measuring processes, absorption and luminescence measurements. Preferably, the measuring point 24 is provided for luminescence measurement, in particular for fluorescence measurement C, as is schematically outlined in Fig. 8. Monochromatic radiation L_{C1} or L_{C2} from an excitation light source 17C, which is part of the hand-held analysis apparatus 1 which will be explained in more detail below, emerges at the measuring point 24 through the liquid sample that is accommodated in the sample-holding space 31

and contains a fluorescent marker substance, which shortly after excitation by the radiation L_{C1} or L_{C2} exhibits fluorescence F. The light emitted in this case is generally lower in energy, i.e. it has a longer wavelength. Unlike as represented, the detector 18C, which records the radiation power of the fluorescence proportional to the concentration of the fluorescent substance, may also be arranged perpendicularly to the axis of the incident light by means of suitable optical elements that are known to the person skilled in the art. Fig. 8 furthermore indicates with the excitation light beams L_{C1} and L_{C2} that excitation light of different wavelengths may be used for detection of different marker substances. For example, blue light L_{C1} of the wavelength 450 nm and green light L_{C2} of the wavelength 530 nm may be selected for the excitation. Thus, a marker may be used which consists of two dye molecules from the range of rylene dyes, for example perylene and quaterrylene (for example Lumogen® F yellow 170, Lumogen® F pink 285, both available from BASF AG, Ludwigshafen, Germany), or a combination of rhodamine carbonyl derivatives and acridine derivatives (for example ATTO® 612 Q 615 nm and ATTO® 495, 498 nm, both available from ATTO-TEC GmbH, Siegen, Germany, so that the two measurement ranges in the long-wave range can be covered.

As an alternative to the fluorescence measurement, in principle a phosphorescence measurement (with corresponding phosphorescent marker substances) is also conceivable. However, while fluorescence ends shortly after the end of the excitation (usually within one millionth of a second), longer, up to an hour long, afterglow usually takes place in the case of phosphorescence. Besides a luminescence measuring point, an absorption measuring point for concentration determination of particular substances may also be envisaged, although fluorescence measurement has a higher selectivity and greater sensitivity compared with absorption measurement.

The second optical measuring point 25 of the sample-holding element 20 is in the present example designed for measuring the refractive index B, one of the plates 30, 30', namely the plate on the light exit side, comprises a prism structure 25', on the inner side in this section provided as a refractive index measuring point 25, as

is schematically indicated in Fig. 7. This section of the sample-holding element 20 with the prism structure 25' at the measuring point 25 therefore forms, with the corresponding components of the hand-held device 1, the refractometer which may use an LED, which for example emits yellow light L_B of the wavelength 580 nm, in an energy-saving manner as the light source 17B. As an alternative to an LED, for example, a laser diode may also be used as the light source. A CCD sensor may be used as the detector 18B for recording the refraction of the light beam. Since the refractive index is temperature-dependent, the hand-held device 1 furthermore comprises, in order to compensate for the temperature influences, a temperature sensor 14 which, like all the other measuring devices of the hand-held device 1, is connected via a corresponding communication line 33 to the data processing unit 13 of the hand-held device 1.

The sample-holding element 20 of Fig. 1 also shows two further measuring points 26 and 27 for pH measurement A and conductivity measurement D. The measuring point 16 is an optical pH measuring point 26, to which end an indicator dye-containing substrate 26' is introduced at this point in the sample-holding space 31 between the two plates 30, 30' (cf. Fig. 6), from the colour change of which after contact with the liquid to be tested the pH can be optically read. As an indicator dye-containing substrate 26', for example, a section of pH paper may simply be envisaged. The measuring components provided therefor of the hand-held device 1 may provide an RGB LED as the light source 17A, the light L_A of which penetrates past the sample-holding element 20 at the pH measuring point 26, is scattered at scattering devices 18A' and is returned onto the indicator dye-containing substrate 26' at the pH measuring point 26, where only the corresponding colour wavelength is reflected again, which can then be recorded by a colour detector 18A and may be used in order to determine the pH.

All the optical measuring components 17A,B,C and 18A,B,C form the optoelectronic measuring device 12 of the analysis apparatus 1 (see Fig. 9) and may, as Fig. 5 indicates, be arranged in an embedding element 16. Representation of optical elements known for the respective measurements A, B, C, such as filters, lenses, mirrors, etc., is omitted for the sake of clarity. Furthermore, Fig. 5 shows a

signal device 19, which at least forwards the signals recorded by the detectors and sensors 18A,B,C. Unlike as represented, an individual signal device may also be provided for each sensor. The signal device 19 is connected by means of the interface 5' and the communication line 33 to the data processing unit 13. Not represented is the fact that the light sources 17A,B,C may comprise a corresponding connection to the controller.

In Fig. 9, an accumulator 11 is furthermore represented as an energy source for the electricity supply of all the components via current lines 33'. The connection of the display device 3 arranged in the other half-shell of the housing 2, which is preferably a touchscreen display, and a (micro)USB interface 5 via corresponding communication lines 33. Instead of or in addition to a (micro)USB interface 5, a memory card slot or a radio interface (WLAN, Bluetooth®, etc.) may also be provided for data transmission from or to an external apparatus. The (micro)USB interface may furthermore be used in order to charge the accumulator 11.

The two half-shells, which form the housing 2, may for example be joined to one another by plug-in or screw connections and opened if required, for instance in order to replace the accumulator 11 or other components. To this end, the half-shells may also be connected in an articulated manner on a long side, for example by means of a hinge, so that the plug-in or screw connections only need to be present on the other side.

Unlike as represented, instead of a chargeable accumulator, a battery may also be provided as the energy source, which is fitted in a compartment which comprises contact means for the batteries and is closed by a housing section that can be opened without a tool, in a known manner, for simplified replacement.

In order to measure the conductivity D of the liquid sample, contact strips 22 are arranged, at the end of the sample-holding element 20 that faces away from the handle section 23, on a section 30'', which protrudes beyond the end of the cover plate 30', of the base plate 30. The exposed ends of these contact strips 22 may come in electrically conductive contact after insertion of the sample-holding

element 20 into the analysis apparatus 1 with corresponding contact elements 15 of the analysis apparatus 1 (cf. Fig. 4), so that an AC voltage can be applied by a frequency generator 18D to the measuring ends of the contact strips 22. The measuring ends of the contact strips 22 form the electrodes at the measuring point 27 and are separated from one another by a predetermined measurement section s. Measurement is actually a resistance measurement, from which the conductivity of the liquid may be calculated.

A further conceivable measuring point of the sample-holding element 20 could be a germ measuring point. An example of germ measurement is represented in Fig. 10. In this case, the venting channel 28 is placed on the sample-holding element 20 in such a way that the air exit opening 29 does not lie in the handle section 23 but forms a measuring point, which is connected by means of a gas communication device 85 to one or more gas sensors, an “electronic nose”. Optionally, the venting channel may comprise cross-sectional variations or a bypass air feed line in order to improve the supply of the molecules present in the vapour phase of the liquid of the electronic nose. The analysis apparatus may to this end, for example, comprise a fan device. As an alternative to the venting channel 28, the existing filling gap may also be used as a germ measuring point for the “electronic nose”. Another approach could be a germ measuring point in which at least one of the plates 30, 30’ comprises a section of a gas-permeable membrane, by which the liquid is retained but through which volatile compounds can pass and thus reach the “electronic flows”. These volatile organic compounds are emissions of bacteria, or germs. An “electronic nose” exists, for example, of sensors coated with different conductive polymers that react specifically for different volatile compounds by changing their electrical resistance in a characteristic way upon contact with these compounds.

As an alternative to the “electronic nose”, germs may also be recorded by means of a luminescence measuring point if a luciferin/luciferase mixture, which reacts with adenosine triphosphate, which occurs in any living cell, is added to the liquid. The light given off may likewise be measured with the luminometer, and is a measure of the microbiological contamination of the liquid.

Depending on the composition of the liquid, a UV absorption measurement may be envisaged as a further method of germ determination, since nucleic acids absorb in the UV range.

The plates 30, 30’ are transparent at least to the corresponding wavelength at least in the region of the measuring points 24, 25, 26, at which optical measuring sensors are used - for the sake of simpler manufacture, the plates 30, 30’ are made entirely of transparent material, which may be glass, preferably quartz glass, or a transparent plastic. Transparent plastics such as PMMA are particularly suitable. The person skilled in the art knows suitable plastics which may be produced in a suitable way simply by 3D printing or extrusion.

Besides the desired transparency, the plastic should be chemically resistant, at least for the duration of the sampling and analysis, to the constituents of the liquid to be received and preferably also, if the sample-holding element 20 comprises a conductivity measuring point, electrically insulating. If the plastic is not electrically insulating enough, the contact strips 22 may be embedded in an insulating material, except for the sample-holding space 31. A transparent plastic which is a good insulator and resistant to aqueous solutions of neutral salts and oxidising agents, as well as to many oils and fats. However, polycarbonates are not resistant to chlorinated hydrocarbons and alkalinely aqueous solutions, amines and ammonia. Another transparent plastic is polymethyl methacrylate, which is resistant to acids, alkalis of medium concentration, petrol and oil, but not to ethanol, acetone and benzene. Polysulfone is likewise transparent in the visible range, but not resistant to ketones, aromatics, chlorinated hydrocarbons and polar solvents. Polymethylpentene has a very high transparency even in the UV range, but is not chemically resistant in the long term to ketones or chlorinated solvents.

The handle section 23, which protrudes at least partially from the analysis apparatus 1 when the sample-holding element 20 is received in the analysis apparatus, may be opaque - as a coloured section of at least one of the plates 30, 30’ or as an attached handle section made of suitable material, for example a plastic. Preferably,

the handle section 23 may be coloured black in order to prevent or minimise stray light incidence. It is, however, also conceivable to characterise different sample-holding elements 20, which differ in type or intended use, by differently coloured and/or shaped handle sections 23.

Furthermore, markings may be applied on the handle section 23 or at other positions of the sample-holding element 20, in order to show and facilitate correct insertion of the sample-holding element 20 into the analysis apparatus 1 for a user. For the same purpose, the sample-holding element 20 may have a rear section 6 which is asymmetrical, in relation to the longitudinal axis of the sample-holding element 20, on the end (with the contact strips 22) facing away from the handle section 23, so that the sample-holding element can only be inserted correctly as far as a stop in the analysis apparatus 1 in one orientation according to the key-lock principle, so that the measuring points 24, 25, 26, 27 can communicate with the respective measuring devices.

Of course, embodiments differing in shape and arrangement from the examples represented also fall within the protective scope of the invention. For instance, a sample-holding element may also have a shape differing from the approximately rectangular shape; this shape, however, is favourable for space-saving arrangement of the measuring points and of the components required for the measurement in the analysis apparatus.

Of course, an embodiment of the analysis device set according to the invention, in which a sample-holding element 20 can be received directly into a correspondingly dimensioned recess of an analysis apparatus 1, is conceivable. According to the invention, however, an insertion device 8, as may be seen in Figs. 2 to 5, 9 and 10, is advantageously provided for this.

The insertion device 8 is fastened in a detachable manner in the analysis apparatus 1 so that it may be replaced if required. The insertion device 8 consists of a shell section 82 which extends inside the analysis apparatus 1, and a flange section 83, which touches, on the outside, against an edge of the housing 2 of the analysis

apparatus 1. In the flange section 83, there is a slot-like insertion opening 9 from which the recess 9'' for the sample-holding element 20 extends through the shell section 82. The latter comprises, as may be seen in Fig. 3, a rear section 7 corresponding with the rear section 6 of the sample-holding element 20. Likewise corresponding with the sample-holding element 20 and also with the measuring devices in the analysis apparatus 1 are openings or transparent sections, as optical communication facilities 81, 81', 81'' in both sides of the otherwise opaque, preferably black, shell section 82 which is in this case also intended to prevent or reduce stray light effects.

In the flange section 83, which is also represented in section in Fig. 2a, a cover plate 4 holds a sealing lip 9' made of silicone on the insertion opening 9. Bores 32, which allow the cover plate 4 to be screwed on the flange section 83, extend through the cover plate 4 and the section parallel thereto of the flange section 83. Unlike as represented, the bores 32 in the flange section 83 may also be through-bores so that the cover 4 can be fastened in a releasable manner by means of screws not only on the flange section 83, but also on the edge of the housing 2 of the analysis apparatus 1. As an alternative, the insertion device 8 may also be designed for insertion/latching into the analysis apparatus 1.

The sealing lips 9' prevent contamination of the recess 9'' in the shell section 82. The insertion device 8 itself in turn prevents the interior of the analysis apparatus 1 from being impurified or contaminated.

Furthermore, the analysis apparatus 1 may comprise an openable cover (not shown), with which the insertion opening 9 may additionally be covered. Such a cover may also be closed when the sample-holding element has been inserted, so that in this case stray light incidence may be prevented and coloration of the handle section may therefore be obviated.

Furthermore, on the end facing away from the flange section 83, the insertion device 8 may comprise electrical bridging elements in or on the the section 30'' of the sample-holding element 20 with the contact strips 22 when the sample-holding

element 20 has been inserted into the recess 9'' of the insertion device 8, which establishes electrical contact between the contact strips 22 of the sample-holding element 20 and the contact elements 15 of the frequency generator 18D, or the insertion element 8 comprises a widening 84 shaped as a socket there, into which the contact elements 15 designed in a plug-like manner of the frequency generator 18D can be received, so that direct electrical contact can be established between the contact strips 22 of the sample-holding element 20 and the contact elements 15 of the frequency generator 18D.

An alternative embodiment of a sample-holding element 20 is shown in Fig. 11. In addition to the already described measuring points 24, 25, 26 and optionally 29 for conductivity, refractive index, pH and optionally germ measurement, the sample-holding element 20 in this example also comprises a nitrite measuring point 26N, which is a predetermined section between the plates 30, 30', in which a nitrite-reactive substrate 26N' that carries out a, for example, photometrically detectable reaction, quantifiable with calibration, with nitrite is introduced, so that, in a similar way to the pH measuring point 26, a colour change may be optoelectronically recorded, by which the presence and amount of nitrate in the sampled liquid may be detected.

The sample-holding element 20 of Fig. 11 also differs in the type of prism structure, used for determining the refractive index, of the measuring point 25. While the prism structure 25' described in connection with the example according to Fig. 7 may consist of a plurality of structures with a triangular profile in an adjacent arrangement, for example pyramidal or tetrahedral structures, or alternatively triangular profiles extending parallel on the inner side of the plate 30, 30' on the light exit side, the refractive index measuring point 25 of the sample-holding element 20 of Fig. 11 comprises a Fresnel lens structure 25'' as a special case of a prism structure, which - likewise on the inner side of the plate 30, 30' on the light exit side - consists of a row of ring-shaped steps.

Fig. 12 shows an insertion element 8 that matches with the sample-holding element 20 of Fig. 11 and is inserted into the embedding element 16 of an analysis apparatus

(not represented further). In contrast to the example shown in Fig. 5, the insertion element 8, in a manner corresponding to the additional nitrite measuring point 26N of sample-holding element 20 as shown in Fig. 11, comprises, as optical communication facilities 81N, a further opening or a transparent sections in two sides of the otherwise opaque, preferably black, shell section 82, in order to prevent or reduce stray light effects in this case as well. In order to measure the nitrite content, a measurement optical element 17AN, 18AN, which may correspond to the measuring device 17A, 18A for the pH measurement, is installed at a corresponding point in the embedding element 16. In this case as well, an RGB LED as a light source 17AN and a colour detector 18AN may be used.

The signal also recorded by the sensor, or colour detector 18A, is transmitted by the signal device 19 via the interface 5' the communication line 33 to the data processing unit.

The measuring components of the refractometer may remain unchanged and, for example, be formed by an (LED or laser diode) light source 17B and a CCD sensor 18B.

One essential point of the invention is that the concentration of the manufacturing medium, or cooling lubricant, in the emulsion exhibits fluorescence after excitation with light of a suitable wavelength by means of an internal marker substance, i.e. it comprises a dye. In addition, the conductivity value, the pH and the refractive index, from which conclusions may also be drawn about the concentration, are at the same time measured with the individual sample-holding element in a single analysis apparatus, which is designed as a portable hand-held device, with a single sampling in one measuring process.

Unlike large static analysis systems, which although they can also record a large characteristic data bandwidth, they can operate economically only a large number of samples of the same type, the analysis device set according to the invention also allows economical use with different special emulsions which are produced only in a limited scope, for instance in small batches, for processing of which there are

particular requirements. Thus, the analysis apparatus, the luminometer of which comprises lasers with (at least) two different excitation wavelengths may be used for concentration recording not only for a conventional cooling lubricant to which a marker substance has been added, or a corresponding emulsion, but also for a so-called “two pack system”. In this case, a booster additive is added to a conventional cooling lubricant emulsion for the purpose of increasing performance during manufacture, usually at a concentration of less than about 5wt%, which is the case in particular when, for example, small batches with particular quality requirements need to be incorporated into the standard manufacturing process. The conventional cooling lubricant in use is then insufficient in terms of performance, so that the machine tool(s) would consequently need to be upgraded for another cooling lubricant with higher performance capability, which would lead to an increased diversity of cooling lubricants and would be uneconomical. In such cases, therefore, the booster additive, which imparts advantageous additional properties of the cooling lubricant, for example in terms of dispersibility, wear protection and/or friction coefficient modification, is to the conventional cooling lubricant. When a booster additive is added, however, it is important to check the concentration thereof for quality assurance, in which case, however, only the constituents contained in the booster additive and not those of the cooling lubricant should be recorded.

To date, it has been possible to carry out booster concentration measurements only elaborately in the laboratory by means of infrared spectroscopy and X-ray fluorescence analysis. It has now surprisingly been found that, with a suitable selection of the marker substance, it is possible to “dope” the booster in such a way that this marker substance does not “diffuse” into the base cooling lubricant. In theoretical terms, therefore, two different emulsion systems essentially exist in parallel, the concentration determination of the base cooling lubricant being carried out by means of a first marker substance and the concentration determination of the booster additive being carried out by means of the second marker substance. An unequivocal concentration determination of the booster additive is therefore also possible in situ, which it has not previously been possible to do.

Of course, the concentration determination of the cooling lubricant and of the booster additive, to each of which a marker substance has been added, in an emulsion by means of the fluorescence measurement of the different marker substances may also be carried out by analysis devices other than the analysis device set according to the invention - although the latter advantageously economically offers rapid analysis directly in situ.

A measuring process that can be carried out with the analysis device set according to the invention may, for example, be carried out as follows:

After the analysis apparatus 1 is turned on, which may be done in a conventional manner by keeping a colour-marked button on the housing 2 depressed, the touchscreen display 3 becomes active - a control light next to the button may optionally also light up - and a selection menu appears on the display 3 with various liquid media, in particular cooling lubricant emulsions, that can be tested, which are deposited in a database that is stored in the data processing unit or on a storage medium (for example a fixed or replaceable storage medium) connected to it. The liquid to be tested may be selected by touching on the touchscreen display 3.

The sampling of the liquid to be tested may be carried out by immersing the sample-holding element 20 with the filling opening in the liquid - or it is sufficient to touch the liquid surface with the filling opening - the sample-holding space 31 being filled with the liquid by the capillary effect. The time taken for this is usually a few seconds and may vary as a function of the selected dimensions of the sample-holding element 20, until the sample-holding space 31 is fully filled with the liquid by the capillary effect, in which case air that is present may escape through the venting channel 28.

The cooling lubricant emulsion, or the liquid, should be mixed well during the sampling. The liquid is therefore optionally to be mixed thoroughly before the sampling in order to ensure a homogeneous distribution of cooling lubricant in the emulsion. As an alternative to immersion or holding on the liquid surface, a pipette or a similar sampling means may also be used in order to take a sample of the liquid,

which is then introduced at the filling opening into the sample-holding space 31 of the sample-holding element 20. If liquid adheres on the outer surface of sample-holding element 20 as a result of the immersion or filling, these or other impurities are removed before inserting the sample-holding element 20 into the analysis apparatus 1.

The sample-holding element 20 held at the handle section 23 is inserted with the section 30'' forward with the insertion opening 9 into the recess 9'' designed as a measuring channel. The sealing lip 9' at the insertion opening 9 prevents contamination of the measuring channel, which is enclosed by the shell section 82 of the insertion device 8, which may be replaced if required, which prevents the interior of the analysis apparatus from being able to be contaminated.

Upon completion of the insertion process, when the contact strips 22 on the extended section 30'' of the sample-holding element 20 contact the contact elements 15 of the analysis apparatus 1, the measuring process is automatically started. If automatic starting of the measuring process is not desired, a user input may also be provided for this, for instance pressing a corresponding message displayed on the touchscreen display 3.

After the end of the measurement(s), a prompt to remove the sample-holding element 20 is displayed on the touchscreen display 3. Once this has been done, the measurement values are displayed. The sample-holding element 20 designed as a single-use test strip may be sent for disposal. It is in principle conceivable, albeit uneconomical, to separate the two plates 30, 30', which constitute the sample-holding element, from one another in order to clean the interior and replenish the pH indicator substrate.

The measurement values may be stored in the data processing unit 13 and/or in a storage medium connected to it. The measurement values may furthermore be transmitted by means of a wireless radio connection, for example according to the Bluetooth® standard, to an external data processing device, or a memory. To this end, a correspondingly labelled field is displayed on the touchscreen display 3, by

actuation of which a preset radio connection is established and the measurement values are transmitted. After the end of the data transmission, this connection is automatically disconnected or may be terminated by another user input.

The turning off of the analysis apparatus 1 may, like the turning on, require pressing of the button for a predetermined time or until the control light is turned on; however, automatic turning off may also be carried out according to a set timer.

With the analysis apparatus 1, it is not only possible to analyse the known liquids which are deposited in the database, but also calibration may be carried out and new liquids/cooling lubricants may be trained, which are then added to the database.

For calibration, a correspondingly labelled field is to be actuated by the user in the start menu on the touchscreen display 3, whereupon a calibration menu is opened, which comprises corresponding control fields for calibration of the analysis apparatus for measurement of the testable parameters refractive index, pH and conductivity. For calibration purposes, the analysis device set comprises different calibration solutions, for example in pipette bottles, which are provided in a separate box.

The calibration menu furthermore comprises correspondingly labelled control fields, by actuation of which new liquids/cooling lubricants labelled with a marker substance may be trained, or media already read in may be recalibrated. To this end, corresponding fluorescence-labelled liquids/cooling lubricants are required. For recalibration, the analysis device set may provide a demonstration solution having a fluorescence-labelled cooling lubricant.

An analysis device set according to the invention, which is provided for analysis of cooling lubricant or cooling lubricant emulsions, may be designed for the following measurement ranges:

- refractive index of from 1.333 to 1.38 (0 to 30 Brix)
- pH of from 7 to 10
- conductivity of from 0.2 to 6 mS/cm

- cooling lubricant concentration in emulsion of from 0 to 15 wt%, or at least in the range of from 0 to 10 wt%, optionally of from 0 to 5 wt %

For a different liquid, the analysis device set may also be designed for different measurement ranges.

LIST OF REFERENCES

1	analysis apparatus	19	signal device
2	housing	20	sample-holding element
3	display device	22	contact strip
4	cover	23	handle section
5,5’	external/internal interface	24	measuring point for fluorescence measurement (concentration)
6, 7	rear section	25	measuring point for refractive index measurement
8	insertion device	25’	prism structure
81,81’,81’’,81N	communication facility	25"	Fresnel lens structure
82	shell section	26	measuring point for pH measurement
83	flange section	26’	indicator substrate
84	socket/opening for contact	26N	nitrite measuring point
85	gas communication facility	26N’	nitrite-reactive substrate
9	insertion opening	27	measuring point for conductance measurement
9’	sealing lip	28	channel
9"	recess	29	air exit opening
11	accumulator	30,30’	plates
12	optoelectronics	30’’	extended plate section for contact strip
13	data processing unit	31	sample-holding space

14	temperature sensor	32	bore
15	contact element	33	communication line
16	embedding element	33’	energy supply line
17A,B,C,AN	light source		
18A,B,C,AN	detector, sensor	L	length of filling gap
18A'	scattering device	s	measurement section
18D	frequency generator		
18E	gas sensor		

Patentkrav

1. Prøvemottakselement (20) for en væskeprøve for samtidig analyse av tre eller flere kjemisk-fysiske parametere for væsken ved hjelp av en analyseanordning,

hvor prøvemottakselementet (20) har et prøvemottaksrom (31) som kan fylles med væsken, hvor prøvemottakselementet (20) fordelt over prøvemottaksrommet (31) har minst tre målepunkter (24, 25, 26, 26N, 27) anordnet tilstøtende hverandre, hvor to av målepunktene (24, 25) er et fotonisk målepunkt (24) og et brytningsindeks-målepunkt (25) og hvor det minst ene ytterligere målepunktet er valgt fra gruppen omfattende minst ett pH-målepunkt (26), ett ledningsevne-målepunkt (27) og ett kim-målepunkt;

hvor prøvemottakselementet (20) er et flatt element (20) som i hvert fall i partier er dobbeltvegget og har to planparallelle plater (30, 30') som er anordnet oppå hverandre og er forbundet med hverandre, hvor prøvemottaksrommet (31) er utformet som en spalte flatt mellom de to platene (30, 30');

hvor platene (30, 30') i hvert fall i partier er forbundet med hverandre ved sine render, hvor en åpning i prøvemottakselementet (20) dannes av de ikke forbundne randdelene, og en avstand mellom platene (30, 30') er akkurat så stor at væskeprøven mellom de dobbeltveggene (30, 30') kan underkastes kapillarvirkningen,

karakterisert ved at

målepunktet (25) for brytningsindeksmålingen har en prismestruktur (25', 25'') ved én av platene (30, 30') i et område forbestemt for dette formålet, hvor platene (30, 30') i det forbestemte området er transparente for bølgelengdene som anvendes for brytningsindeksmålingen, hvor prismestrukturen (25', 25'') tilveiebringer flatepartier som er vinklet med hensyn til plateplanet, der innfallende lysstråler brytes motsvarende.

2. Prøvemottakselement (20) ifølge krav 1,

karakterisert ved at

platene (30, 30') langs minst én side, fortrinnsvis langs en langside, ikke er forbundet med hverandre, slik at en fylleåpning eller en fyllespalte med en lengde (L)

for væsken tilveiebringes.

3. Prøvemottakselement (20) ifølge krav 1 eller 2,
karakterisert ved at

det flate elementet (20) i det minste delvis består av lysgjennomskinnelig glassmateriale eller et gjennomsiktig plastmateriale.

4. Prøvemottakselement (20) ifølge minst ett av kravene 1 til 3,
karakterisert ved at

lengden til én av de to platene (30) i hvert fall ved én ende er større enn lengden til den andre platen (30') og har et parti (30'') der minst to kontaktstriper (22) for påtrykking av spenning er anordnet, som strekker seg inn i prøvemottaksrommet (31) og ender der i en avstand fra hverandre svarende til en målestrekning (s) som danner målepunktet (27) for måling av ledningsevne.

5. Prøvemottakselement (20) ifølge krav 4,
karakterisert ved at

det flate elementet (20), ved en annen ende som vender vekk fra enden med kontaktstripene (22), er utformet som et håndtaksparti (23) for håndtering av prøvemottakselementet (20), og ved at en fluidbane fortrinnsvis strekker seg fra fylleåpningen eller fyllespalten med lengde (L) langs målepunktene (24, 27, 25, 26, 26N) til en luftekanal (28) som ender ved en luftutgangsåpning (29) på yttersiden av det flate elementet (20).

6. Prøvemottakselement (20) ifølge minst ett av kravene 1 til 5,
karakterisert ved at

- det fotoniske målepunktet (24) er et luminescensmålepunkt (24), hvor platene (30, 30') i et forbestemt første parti er transparente for eksitasjons- og utstrålingsbølgelengdene til den planlagte luminescensmålingen, og/eller

- målepunktet for pH-målingen (26) har et indikatorfargestoffholdig substrat (26') som er anordnet i et forbestemt andre parti mellom de to platene (30, 30'), og/eller

- prismestrukturen (25') dannes av minst én, fortrinnsvis flere, tilstøtende anordnede strukturer som har et trekantprofil eller en Fresnel-linsestruktur (25'') som har en rekke av ringformede trinn, som er tilveiebragt ved et forbestemt tredje parti av én av de to platene (30, 30'), og/eller

- den gruppen som det minst ene ytterligere målepunktet er valgt fra i tillegg omfatter et nitritt-målepunkt (26N) som har et nitritt-reaktivt substrat (26N') som er anordnet i et forbestemt fjerde parti mellom de to platene (30, 30').

7. Analyseanordningssett for samtidig analyse av minst tre forskjellige kjemisk-fysiske parametere for væsker;
hvor analyseanordningssettet omfatter

- et analyseapparat (1) utført som et håndapparat med et hus (2) og med en fremvisningsanordning (3), samt

- minst ett prøvemottakselement (20) for en væskeprøve,
karakterisert ved at

prøvemottakselementet (20) er et prøvemottakselement (20) ifølge minst ett av kravene 1 til 6,

og analyseapparatet (1) har en optoelektronisk analyseanordning (12) og en databehandlingsenhet (13) som er kommunikativt forbundet med analyseanordningen (12) og fremvisningsanordningen (3),

hvor den optoelektroniske analyseanordningen (12) har minst tre måleanordninger (15, 17, 18) anordnet tilstøtende hverandre, hvis anordning korresponderer med anordningen av målepunktene (24, 25, 26, 26N, 27) på prøvemottakselementet (20).

8. Analyseanordningssett ifølge krav 7,
karakterisert ved at

analyseapparatet (1) har en innsettingsanordning (8) for mottak av

prøvemottakselementet (20), som er løsbart anordnet i huset (2) og har en innsettingsåpning (9) som munner ut i en utsparing (9") for mottak av prøvemottakselementet (20) som er utformet for å korrespondere med det, hvor innsettingsanordningen (8) har en optisk, elektronisk eller optoelektronisk kommunikasjonsinnretning (81, 81', 81", 81N) som korresponderer med anordningene av måleanordningene (15, 17, 18) og målepunktene (24, 25, 26, 26N, 27) i avhengighet av typen til det respektive målepunkt (24, 25, 26, 26N, 27).

9. Analyseanordningssett ifølge krav 7 eller 8,
karakterisert ved at

innsettingsanordningen (8) har et flensparti (83) med innsettingsåpningen (9) og et kappeparti (82) som er løsbart anordnet i huset (2), avgrenser utsparingen (9") og har de optiske, elektroniske eller optoelektroniske kommunikasjonsinnretningene (81, 81', 81", 81N), som er dannet av partier av gjennomsiktig materiale og/eller av åpninger i kappepartiet (82), som ellers er laget av opakt materiale.

10. Analyseanordningssett ifølge minst ett av kravene 7 til 9,

hvor to av måleanordningene (15, 17, 18) er en fotonisk måleanordning, fortrinnsvis en luminescens-måleanordning (17C, 18C), og en brytningsindeks-måleanordning (17B, 18B) og hvor den minst ene ytterligere måleanordningen (15, 17, 18) er valgt fra gruppen omfattende minst én pH-måleanordning (17A, 18A), én ledningsevne-måleanordning (15, 18D), én nitritt-måleanordning (17AN, 18AN) og én måleanordning (18E) for deteksjon av kimbelastningen,

- luminescens-måleanordningen (17C, 18C), brytningsindeks-måleanordningen (17B, 18B), pH-måleanordningen (17A, 18A) og nitritt-måleanordningen (17AN, 18AN) hver har en lyskildeenhet (17A, 17B, 17C, 17AN) og en deteksjonsenhet (18A, 18B, 18C, 18AN) som er anordnet i huset (2) på begge sider av de motsvarende målepunktene (24, 25, 26, 27, 26N) til prøvemottakselementet (20) mottatt i analyseapparatet (1),

- ved at analyseapparatet (1) har en temperaturmåleanordning (14) som er forbundet med databehandlingsenheten (13);

- ledningsevne-måleanordningen (15, 18D) har en frekvensgenerator (18D) med kontaktelementer (15), som når prøvemottakselementet (20) er anordnet i analyseapparatet står i elektrisk kontakt med de minst to kontaktstripene (22) på prøvemottakselementet (20);

- måleanordningen (18E) for deteksjon av kimbelastningen er minst én mikroelektronisk gassensor (18E) som står i forbindelse med prøvemottaksrommet (31) via en forbindelsesledning.

11. Analyseanordningssett ifølge minst ett av kravene 8 til 10,
karakterisert ved at

flenspartiet (83) til innsettingsanordningen (8), i en analyseanordning, der innsettingsanordningen (8) er innsatt i huset (2), er anlagt på yttersiden mot en rand av huset (2) og innrammer en dekkplate (4), der innsettingsåpningen (9) er dannet, som er avtettet av en tetningsleppe (9') som holdes i flenspartiet (83) av dekkplaten (4), hvor dekkplaten (4) er løsbart festet i flenspartiet (83).

12. Analyseanordningssett ifølge krav 10 eller 11,
karakterisert ved at

innsettingsanordningen (8) har kontaktbroer som skaper kontakten til kontaktelementene (15) til analyseapparatet (1) med de minst to kontaktstripene (22) til prøvemottakselementet (20).

13. Analyseanordningssett ifølge minst ett av kravene 7 til 12,
karakterisert ved at

analyseapparatet (1) har en energikilde, fortrinnsvis en akkumulator (11), som er anordnet i huset (2) og sørger for energiforsyningen til den optoelektroniske analyseanordningen (12), databehandlingsenheten (13) og fremvisningsanordningen (3).

14. Analyseanordningssett ifølge minst ett av kravene 7 til 13,
karakterisert ved at

- fremvisningsanordningen (3), som betjeningsgrensesnitt, er en berøringsfølsom fremvisningsanordning (3),

- databehandlingsenheten (13) har eller er forbundet med et eksternt kommunikasjonsgrensesnitt (5), hvor det eksterne kommunikasjonsgrensesnittet (5) er et stikkontaktgrensesnitt eller et radiogrensesnitt.

15. Fremgangsmåte for samtidig analyse av minst tre forskjellige kjemisk-fysiske parametere for en væske med bruk av et analyseanordningssett ifølge minst ett av kravene 7 til 14,
omfattende de trinn å

- neddykke prøvemottakselementet (20) i væsken eller bringe væskeoverflaten i kontakt med en åpning i prøvemottakselementet (20) som er dannet av de ikke forbundne randdelene, og fylle prøvemottaksrommet (31) til prøvemottakselementet (20) med en væskeprøve ved hjelp av kapillarvirkningen mellom de doble veggene (30, 30') til prøvemottakselementet (20),

- sette prøvemottakselementet (20) helt inn i analyseapparatet (1),

- innlede og gjennomføre minst tre eller flere måleprosesser samtidig ved hjelp av måleanordningene (18A,B,C,D,E,EN) i målepunktene (24, 25, 26, 27, 28, 26N),

- etter at måleprosessene er avsluttet, vise måleresultatene på fremvisningsanordningen (3).

16. Fremgangsmåte ifølge krav 15,
hvor

- forskjellige undersøkbare væsker er deponert i en database som er lagret i databehandlingsenheten eller på et lagringsmedium forbundet med denne, og, før innledningen og gjennomføringen av minst tre eller flere måleprosesser samtidig ved hjelp av måleanordningene (18A,B,C,D,E,EN) i målepunktene (24, 25, 26, 27, 28, 26N), velge væsken som skal undersøkes gjennom en brukerinnmating på fremvisningsanordningen (3).

17. Fremgangsmåte ifølge krav 15 eller 16,
omfattende de trinn å

- automatisk eller etter en brukerinnmating, detektere fullstendig innsetting av prøvemottakselementet (20) i analysapparat (1), og/eller

- etter avslutning av måleprosessene, vise, på fremvisningsanordningen (3), en oppfordring om å fjerne prøvemottakselementet (20) fra analyseapparatet (1), og/eller

- etter at uttak av prøvemottakselementet (20) fra analyseapparat (1) er detektert, vise måleresultatene på fremvisningsanordningen (3) og lagre og/eller overføre måleresultatene.

18. Fremgangsmåte ifølge krav 15 eller 16,
omfattende de trinn å

- kalibrere analyseapparatet (1) for de undersøkbare væskene, som er deponert i databasen, med bruk av kalibreringsløsninger, og/eller

- lese inn nye væsker med kjente kjemisk-fysiske parametere med analyseapparatet (1) og legge til de innleste væskene i databasen.

19. Fremgangsmåte ifølge krav 17 eller 18,
hvor

væsken omfatter minst én markørsubstans som kan påvises ved hjelp av luminescensanalyse, og hvor ett av målepunktene (24, 25, 26, 27, 28, 26N) er et luminescens-målepunkt (24).

20. Fremgangsmåte ifølge krav 19,
hvor

væsken som skal analyseres er en metallbearbeidingsvæske, spesielt et kjølende smøremiddel, spesielt foretrukket en kjølende smøremiddelemulsjon, hvor væsken tilsettes minst én første markørsubstans, som kan påvises ved hjelp av luminescensanalyse, i en forbestemt konsentrasjon.

21. Fremgangsmåte ifølge krav 20,

hvor

væsken omfatter et boosteradditiv og væsken tilsettes minst én andre markørsubstans, som kan påvises ved hjelp av luminescensanalyse, i en forbestemt konsentrasjon, hvor den andre markørsubstansen er forskjellig fra den første markørsubstansen med hensyn til sine luminescensegenskaper.

Fig. 1

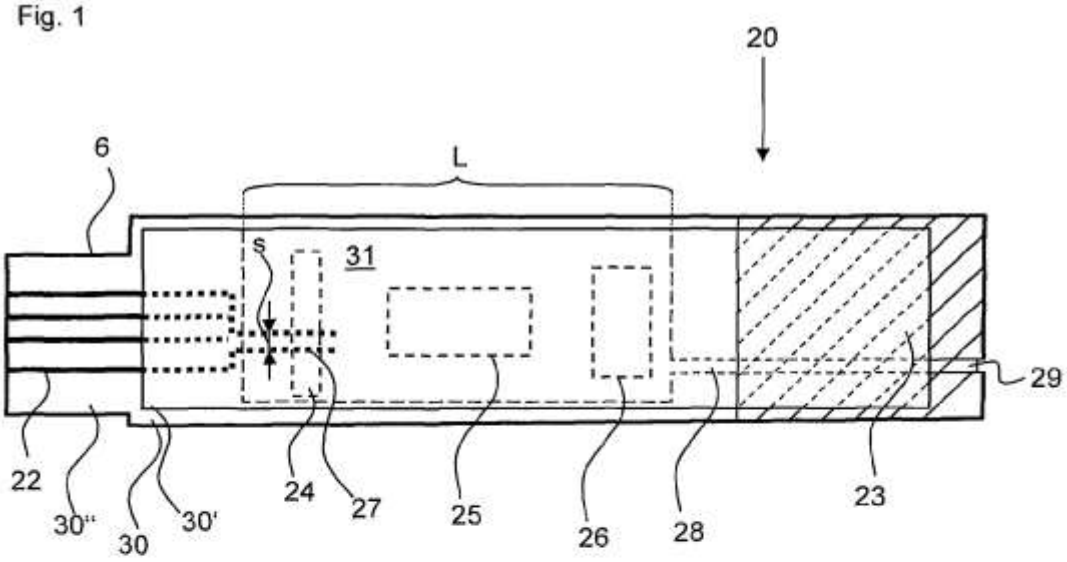

Fig. 2

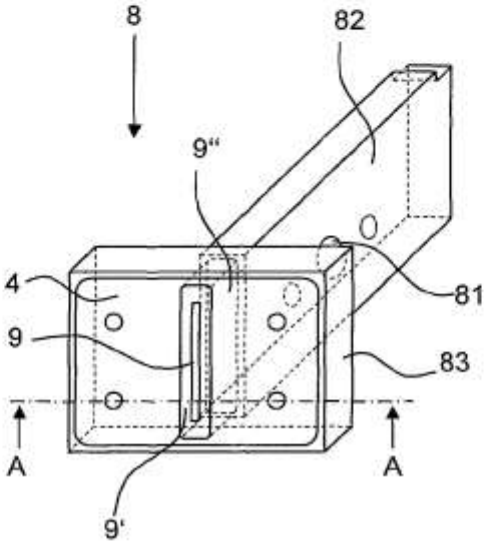

Fig. 2a

A - A

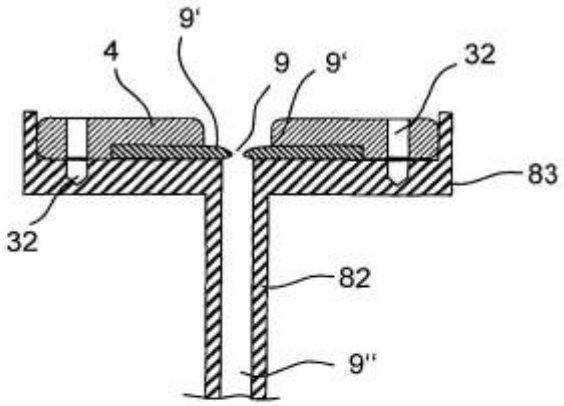

Fig. 3

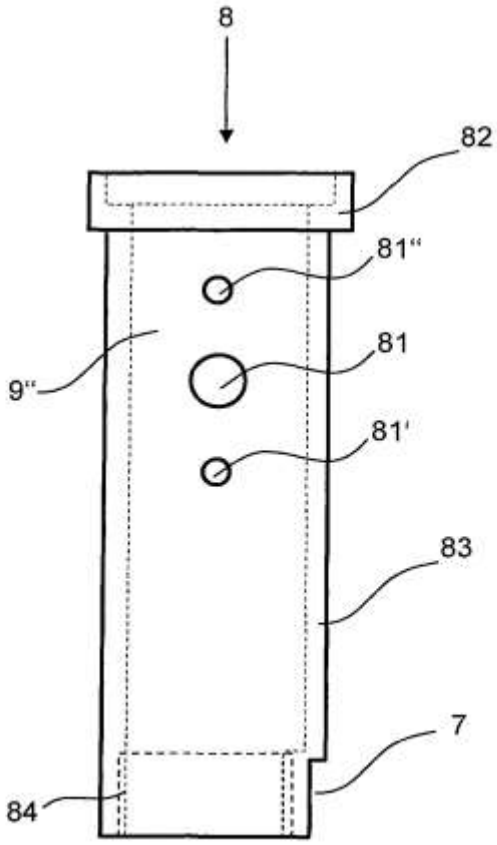

Fig. 4

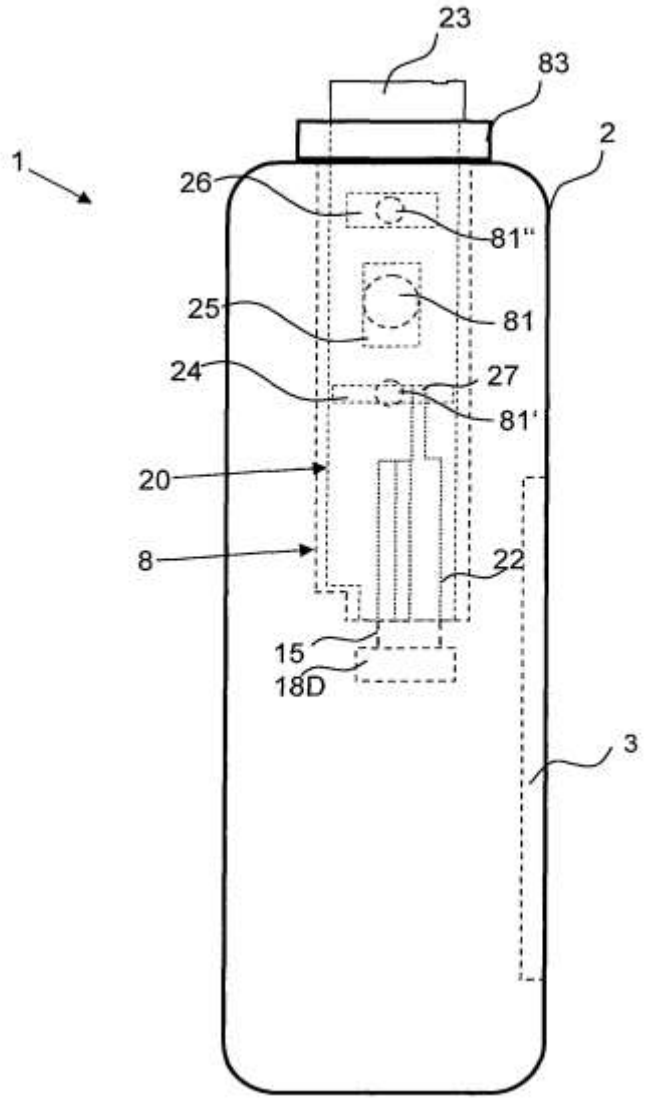

Fig. 5

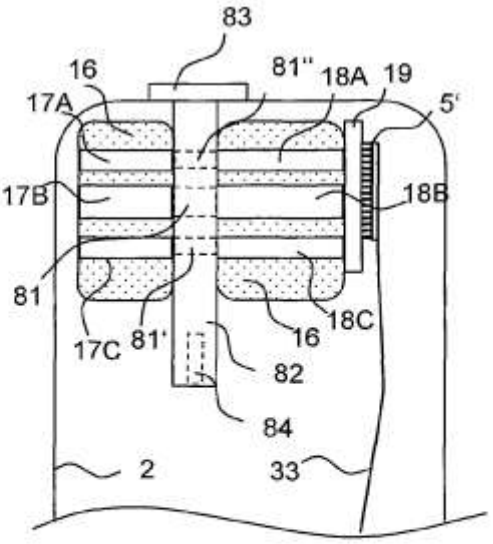

Fig. 6

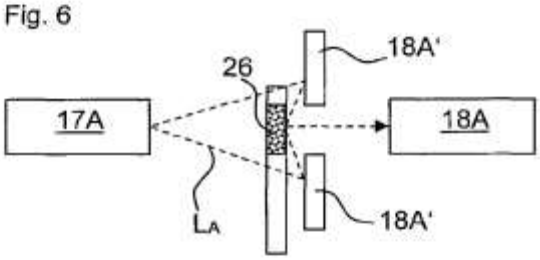

Fig. 7

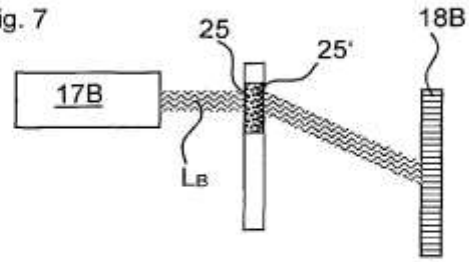

Fig. 8

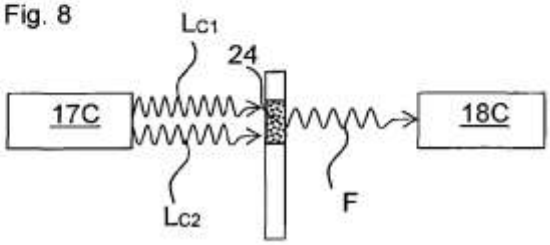

Fig. 9

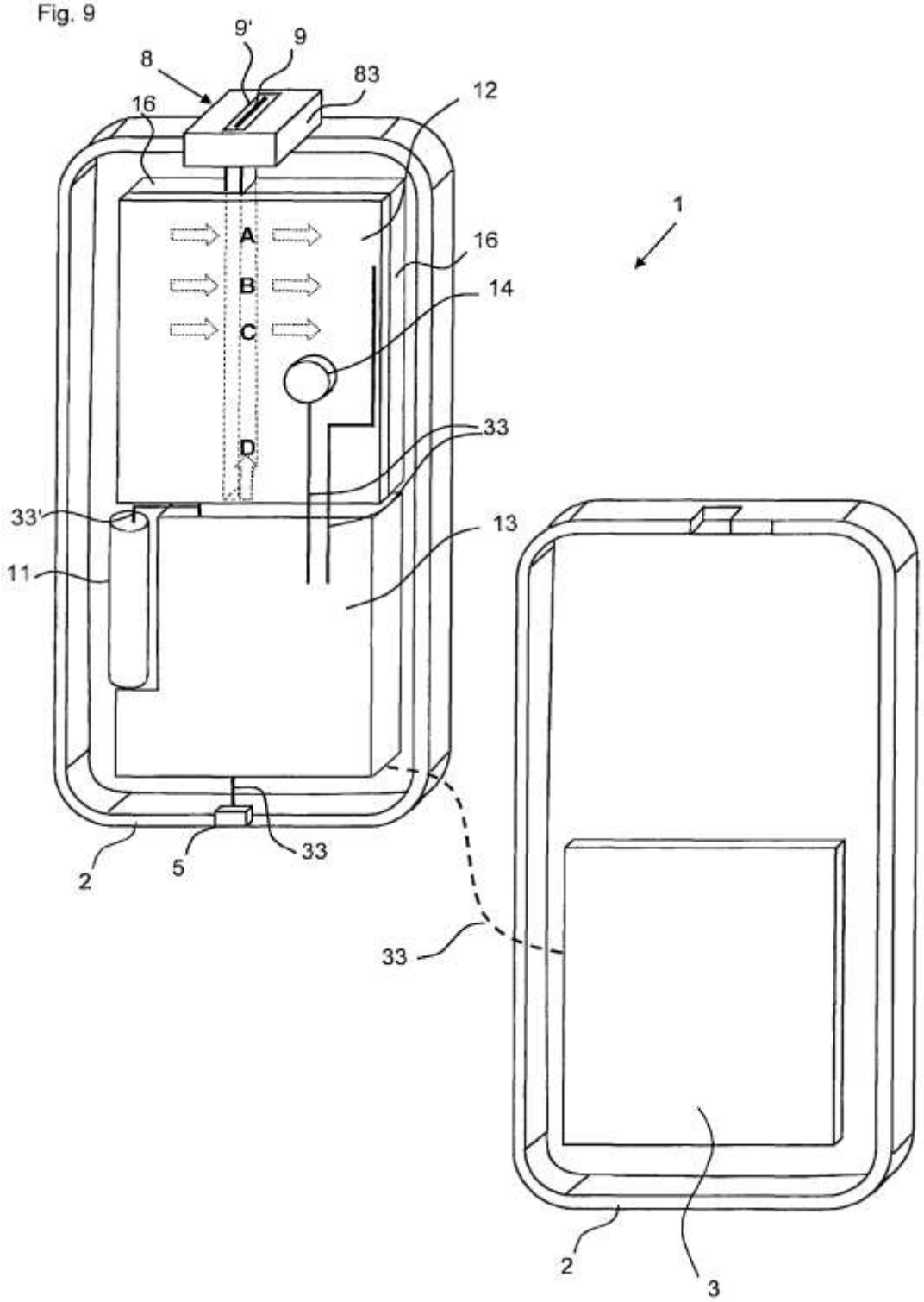

Fig. 10

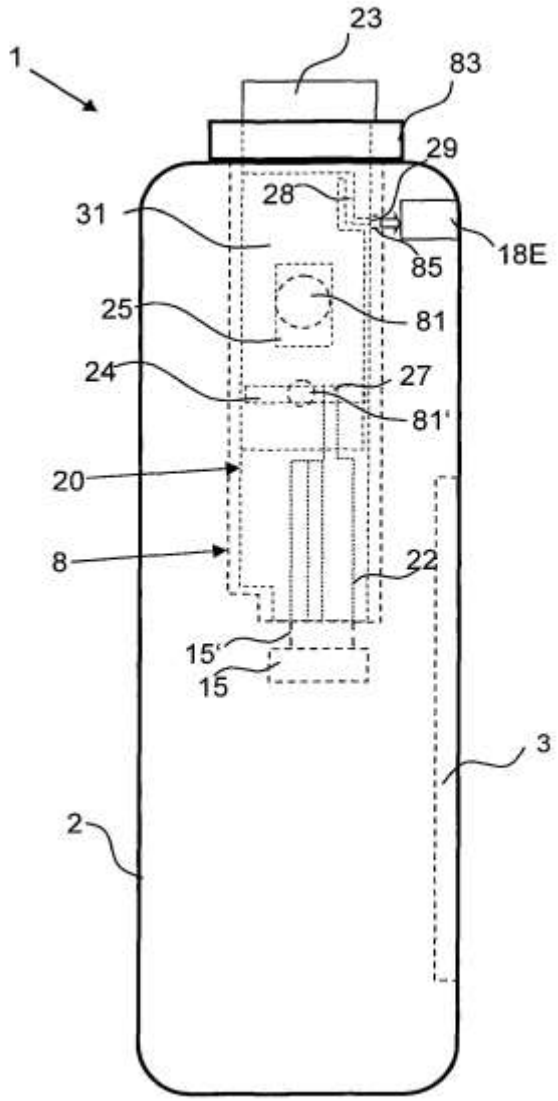

Fig. 11

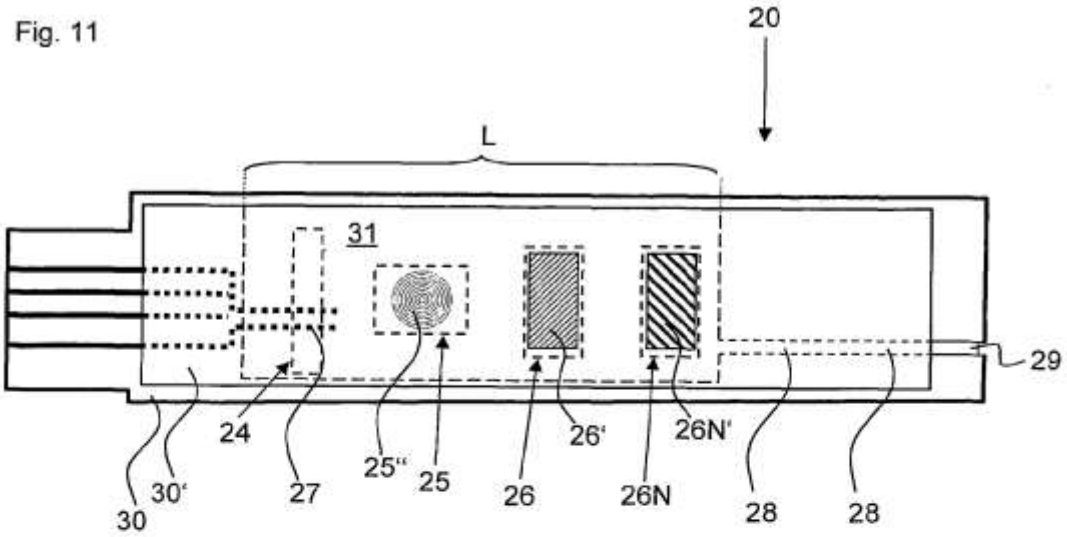
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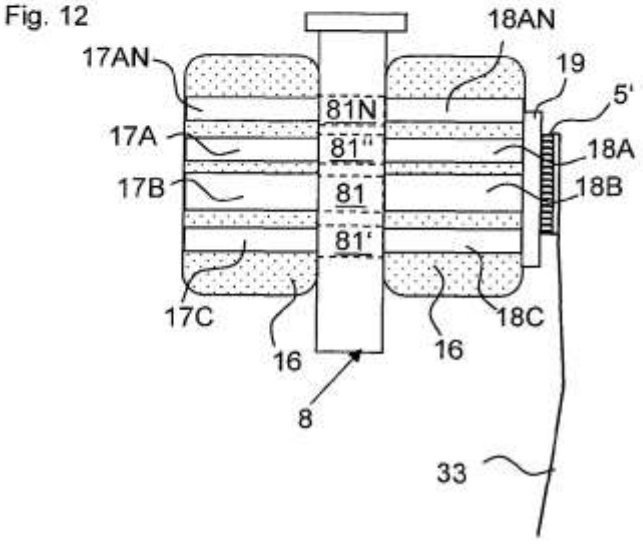
Fig. 12


18AN
17AN
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5'
81N
17A
81''
18A
17B
81
18B
81'
17C
18C
16
16
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33