Matthias Kemmerling, Jens Karl Wegener, Dirk Rautmann,
Jan-Philip Pohl, Eckhard Immenroth, Dieter von Hörsten

Trial Report – Closed Transfer Systems (CTS)

Julius Kühn-Institut (JKI), Bundesforschungsinstitut für Kulturpflanzen
Institute for Application Techniques in Plant Protection

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Julius Kühn-Institut
Bundesforschungsinstitut für Kulturpflanzen

Kontaktadressen/ Contacts
Julius Kühn Institut
Federal Research Center for Cultivated Plants
Institute for Application Techniques in Plant Protection
Messeweg 11/12
38104 Braunschweig
Germany
E-Mail: at@julius-kuehn.de
Telefon +49 (0) 5 31 -2 99 36 51
Telefax +49 (0) 5 31 -2 99 30 12

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Vertrieb
Saphir Verlag, Gutsstraße 15, 38551 Ribbesbüttel
Telefon +49 (0) 5374 6576
Telefax +49 (0) 5374 6577
verlag@saphirverlag.de

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1. Introduction

Closed Transfer Systems (CTS) are closed systems which are used to protect the operator against contamination with undiluted plant production products (PPP) during filling the plant protection equipment. The filling process is carried out as possible without contact to the PPP. A CTS is a universal solution that can be used on a wide range of different sprayer types and sizes. It offers the possibility of using and rinsing different container sizes up to large packages with a single system. Thus, it makes it unnecessary to switch between different dosing aids and allows a separate cleaning at the same time. Due to their design, the system components are easy to clean, even with partial emptying. So far, however, there are only a few available systems on the market and at the moment there is no reliable data about the contribution of CTS to operator protection.

To assess the protective effect of CTS on the operator, a method for measuring operator exposure was developed at JKI. In the context of the predecessor project "EasyFlow", the dermal exposure of an operator during the filling process on a field crop sprayer has already been measured. For this purpose, the system easyFlow (Agrotop GmbH) was used and the exposure was determined using internal and external dosimeters on the whole body in independent experiments. Personal protective equipment used as external dosimeter consisting of a protective overall, protective gloves and protective visor was used in accordance with Good Agricultural Practice. In addition, as internal dosimeters representing the operator's skin, long underwear, cotton gloves and a patch on the neck of the operator were worn. Instead of a real pesticide, a fluorescent tracer dissolved in water was filled into PPP canisters and sealed. The first results showed a significant reduction of the measured exposure of the protective gloves by the use of easyFlow.

In the current project, the existing measurement method was reconsidered and various measures have been implemented to improve the procedure. In addition, the developed methodology was tested and validated in an extended test scope with several operators. The aim was to increase the measuring accuracy and thus the significance of the measured data in order to create a general basis for the examination of operator contamination during the filling process. The main purpose of the project was to get an accepted test method for CTS of different manufacturers. Based on the representative, experimental investigations relevant parameters for the operator protection should be determined. Finally, criteria should be derived, which form a basis for an assessment of CTS.

The project did not aim to assess the used CTS with regard to its reduction of operator exposure and to perform a classification of that specific system. This remains reserved for further testing.

2. Project overview

2.1 General information

The purpose of the considered measurement method is to determine the potential dermal exposure of the operator during the filling process by means of a fluorescence tracer using whole body dosimeters. The task is to determine the amount of fluid that the operator might come into contact with when filling the device under realistic circumstances. The entire process is generally composed of three successive sub-processes, which are carried out consecutively at different times.

1. **Filling experiment with operator**
2. **Washout and sampling**
3. **Sample preparation and sample analysis**

The first step is to carry out the filling experiments. For this purpose, an operator is clothed on the entire body with dosimeters for absorbing the tracer. The outer dosimeters are clothes that are used as personal protective equipment for the application of pesticides (protective overall, protective gloves and protective visor). In addition, inner dosimeters are used. These clothing items are representative of the skin of the operator. The shoes are not recorded. With the dosimeters, the operator carries out a defined filling cycle in each experiment, which involves fillings of several canisters of the tracer solution. To simulate real-life conditions, the filling cycle is carried out on a real field crop sprayer and with sealed canisters. The contaminated dosimeters are removed after completion of the test and separated and light-tight packed in sacks or closed containers.

In the next step, the tracer has to be washed out of the item of clothing to determine the collected tracer quantity in the laboratory. Small dosimeters such as visors, gloves and filter papers can be rinsed with distilled water or swirled on a laboratory shaker in distilled water. The large textile dosimeters are reused several times. They are therefore washed out in a common drum washing machine with demineralized water. The used wash water volume is measured and a sample is taken.

In the last step the samples are analyzed using a spectrofluorometer to determine the tracer concentration in the sample. Since remaining fibers in the samples can clog the flow through cuvette and lead to falsifications of the measurement values, all potentially fiber-loaded samples have to be processed with filters prior to analysis. In the spectrofluorometer, the sample liquid is irradiated with the light of a substance-specific wavelength (excitation wavelength), whereby the molecules of the tracer are excited to fluoresce. The intensity of the light emitted thereby is measured at a further substance-specific wavelength (emission wavelength) and output by the appliance. Since the fluorescence intensity (FI) is linearly related to the concentration of the tracer, it can indirectly measure the concentration of the sample. In order to determine accurately the relationship between fluorescence intensity and concentration, however, a regular calibration is required.

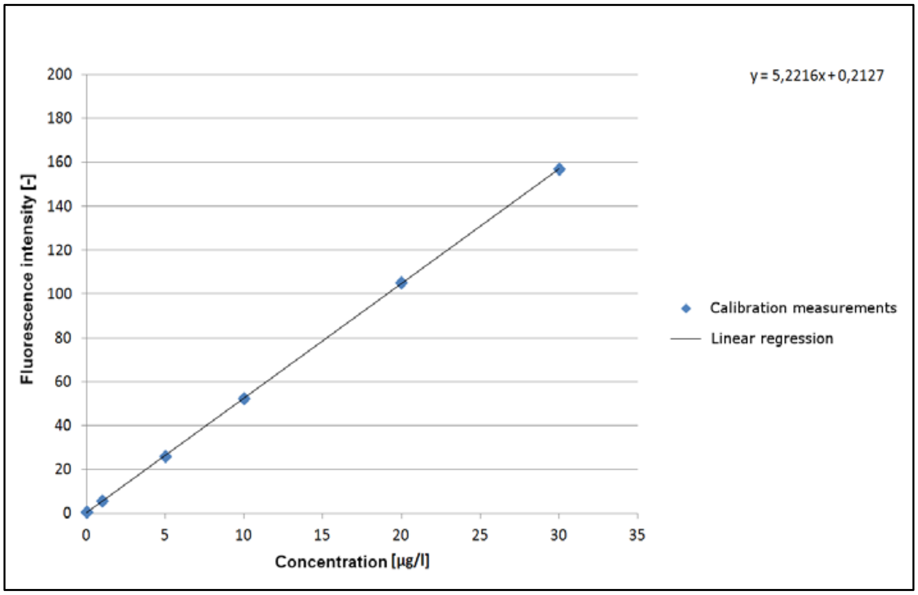

Figure 1: The calibration line is a linear regression from the calibration solution readings and represents the relationship between the flourescence intensity reading and the tracer concentration.

The tracer used for the experiments is a solution of the brilliant yellow fluorescent dye pyranin 120% from Lanxess with a concentration of 5 g / l. The solution is prepared in a large mixing tank and then filled into the canisters and sealed afterwards. Pyranin is non-toxic and can be detected well in very low concentrations. The water solubility of pyranin at 300 g / l (Lanxess Deutschland GmbH 2017) is well above the concentration used here. Thus, a good mixing and a uniform concentration over the total volume can be ensured. However, results from another study showed that the fluorescence degrades under the influence of light over time (Herbst et al. 2006), which is why the storage of the samples is of particular importance.

Figure 2: Pyranin is initially in powder form before being dissolved in water.

2.2 Review of the previous project EasyFlow

The first project to measure exposure "EasyFlow" had a duration of 8 months (01.10.2016 - 31.05.2017) and was carried out together with the Federal Institute for Risk Assessment (BfR) at the Institute for Application Techniques in Plant Protection at the Julius Kühn-Institute (AT). It was examined to what extent the operator contamination can be reduced by using the CTS easyFlow from Agrotop GmbH in comparison to the conventional filling (JKI 2018) The easyFlow system consists of a tank adapter, which can either be mounted on the induction hopper or on the tank of the sprayer, and several canister adapters for connection to the PPP canisters. The canister adapters can be used across all manufacturers with all PPP packages with 63 mm screw thread. The filling volume can be up to 10 liters. (agrotop GmbH 2016) In order to detect the operator contamination, filling experiments were carried out on a field crop sprayer of type Rau D2. With 10 repetitions each, the filling process was examined via the induction hopper and via the dome shaft with easyFlow and also conventionally without easyFlow. All experiments were carried out by the same operator, each with two 5 liter canisters outdoors.

Figure 3: Outdoor experiments with 5 liter canisters of the EasyFlow project (JKI 2018)

The exposure was recorded with six different dosimeters. The operator wore a one-piece work overall and underneath long underwear to cover the body. Protective gloves and underneath cotton gloves were worn on the hands. Furthermore, a protective visor was worn in front of the face and a patch of filter paper on the neck. While the washout of the tracer of visor, filter paper and gloves was done in the analytical laboratory with distilled water, the overalls and underwear were washed out in a common washing machine. In each case, two consecutive washings were run through and samples were taken from the washing water. The first sample was used to determine the tracer content, and the second sample was used to determine a blank value needed to correctly calculate the exposure data.

This blank value ("zero value") is a fluorescence intensity that is measured even if there is no tracer contamination and must therefore be subtracted from the FI measured values. The amount of the blank value mainly depends on the material of the dosimeter. Before the tracer concentration is calculated under consideration of the calibration curve, the measured values have to be checked. The detection limit and the quantification limit of the measurement procedure have to be taken into account in order to obtain reliable results. The detection limit is defined as the sum of the averaged blank value and threefold the standard deviation. Only above this limit it can be confirmed that tracer is found in the sample. However, accurate quantification of the concentration is only possible above the limit of quantification. The limit of quantification is defined here as the sum of the averaged blank value and ninefold standard deviation. For the calculation of the measurement results, this means that measured values below the detection limit are assigned to have the value zero. Measured values that are between the detection limit and the quantification limit are assigned the average value of both limits. After the concentration has been determined with these corrected FI measured values, the absolute tracer mass can be calculated by multiplying with the sample volume.

Ultimately, contamination could not be detected in all dosimeters. For the protective visor as well as for the neck filter, the detection limit was exceeded only in two individual experiments. For cotton gloves, the detection limit was never exceeded.

Only in the case of protective gloves results could be determined above the quantification limit in all variants. The tracer mass found is significantly higher in the variants with easyFlow than in the comparison versions with conventional fillings. In addition, more than twice as high values are measured at the dome shaft compared to fillings using the induction hopper.

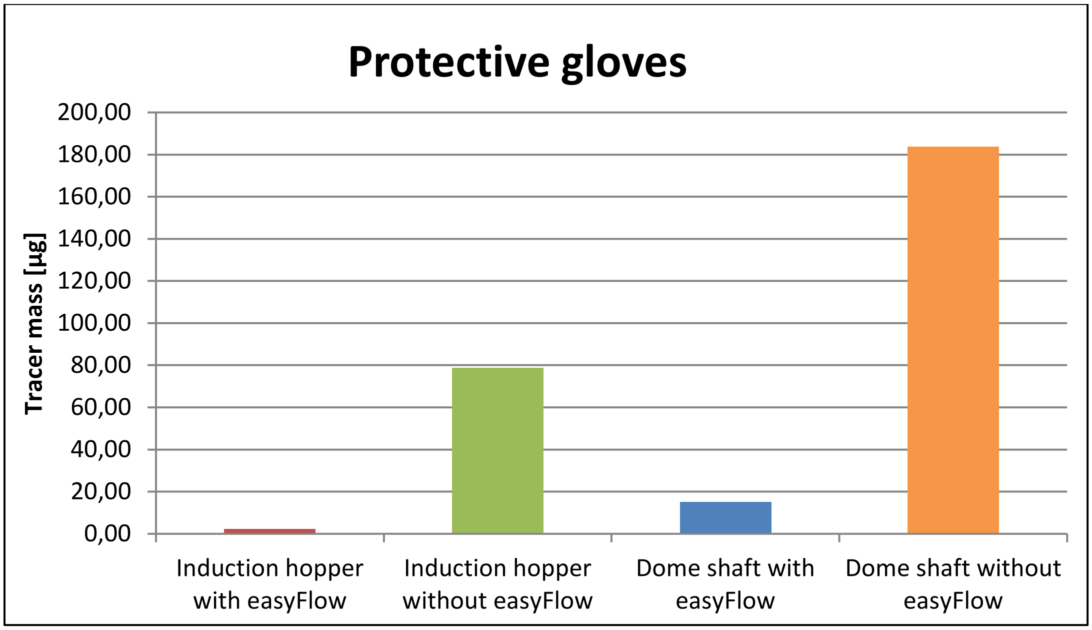

Figure 4: Results (75th percentile) of plant protection gloves for different filling variants (JKI 2018)

The detection and quantification limits of the overalls were relatively high compared to the other dosimeters. Therefore, the measured values were often between detection limit and quantification limit, except for the test variant with easyFlow at the dome shaft. In this variant, the detection limit was not exceeded across all repetitions. In comparison of the two variants without easyFlow higher contamination was found when filling over the dome shaft.

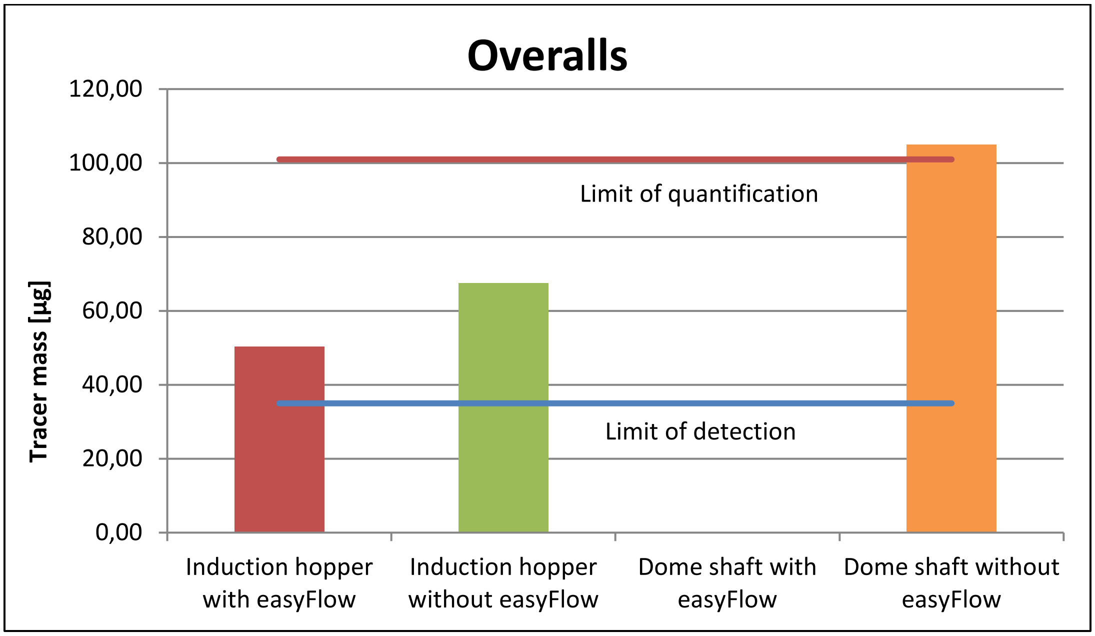

Figure 5: Results (75th percentile) of overalls for different filling variants

In two individual tests on the induction hopper with easyFlow, the measured values of the underwear are above the limit of quantification. In all other variants, all values are below. However, it was found that the determined contaminations occur even if the detection limit on the overall worn to it was not exceeded. Since the underwear was worn under the overall, it can be concluded that the increased values were not caused by the tracer.

A reduction potential with regard to operator contamination by easyFlow could be proven, since at least the plant protection gloves in all variants provided measured values above the limit of quantification. Nevertheless, the results show that the measurement method provides only limited meaningful results in some areas. This is mainly due to the high detection and quantification limits of underwear and especially in the tests concerning protective overalls. Although the use of the washing machine to wash out the body dosimeters has proven to be successful in principle, the high blank values in conjunction with the high water quantities indicate here a potential for optimization. Furthermore, it has been noticed as a debility that all experiments were carried out by the same operator. Since different operators differ in the filling process due to different body size, different handling, etc., the reproducibility of the results may not be guaranteed.

2.3 Innovations of the current project

Representative exposure measurements are enormously time-consuming. Furthermore, the measurement results can show relatively large deviations. This is partly because different frame conditions (e.g., test person, experimental procedure, experimental equipment, environmental conditions, etc.) can cause deviations. On the other hand, due to the complex measuring method, which consists of several process steps, relevant systematic and statistical deviations may occur. The challenge is to develop a measurement method that delivers accurate and reproducible measurement results while capturing as many relevant influencing factors as possible for the selected problem. While the capture of relevant influences is mainly ensured by a suitable test design, measurement accuracy and reproducibility can be increased by optimizing the measurement methodology. The optimization of measures developed in the current CTS project are explained in detail below. One focus was on achieving a low and, as far as possible, stable blank value for all types of dosimeter in order to be able to carry out consistently trouble-free measurements. The scatter of the measured values should be reduced to a minimum, so that the detection and quantification limit are at a low level and thus the smallest possible quantities become quantifiable. Furthermore, the recovery rate should remain at a high level even with low contamination and the reproducibility of the measured data should be increased as much as possible. In order to achieve these results, numerous preliminary studies were carried out considering various aspects.

2.3.1 Washout and sample preparation

The measurement accuracy depends, among other things, on the current sample concentration. Higher concentrations can be measured with lower sensitivity and thus lower measurement noise. Furthermore, the robustness increases against disruptive effects, such as unintentional pollution or cross-contamination of the sample. A conclusion from the predecessor project therefore was, that the water volume of the washing machine should be reduced. In the EasyFlow project the overalls were washed with 60 liters per wash cycle in the program "Jeans" at 60 °C and the underwear with 22 liters of water in the program "Shirts" at 30° C. The washing machine model used at that time (AEG LAVAMAT 61470FL) did not offer a more suitable washing program, which is why in the CTS project was switched to a different washing machine model (Miele W1 Classic). With this model, using the program "Express 20", overalls and underwear can be equally washed at 30 °C. The trapped volume of water was reduced to about 17 liters per washing. Within a total of 20 minutes, the drum is filled twice with water and emptied again. At the end of the wash cycle, the laundry then is spinned intensively to remove the residual water. As a result, the tracer is effectively rinsed out of the textile and removed in a relatively short time.

Figure 6: AEG LAVAMAT 61470FL

Figure 7: Miele W1 Classic

In the EasyFlow project, the washing machine was fed with normal tap water. This corresponds to the intended operation of the machine, was easy to implement and cost-effective in view of the high water consumption. However, the blank value with tap water is higher due to the salts contained in the water and other substances dissolved in the water. Furthermore, temporary fluctuations in tap water quality can lead to uncontrolled blank value fluctuations. Therefore, the new washing machine was preceded by its own demineralizer, which produces from the ordinary tap water demineralized water of constant quality. The water is passed through an ion exchange resin, which retains foreign particles and binds the salts contained in the water. Short hose lengths between this system and the washing machine additionally ensure that no deposits from the water pipe get into the machine. A device for measuring the electrical conductivity and a ball valve for taking water samples allow a constant control of the water quality.

Figure 8: Demineralizer AFT Pro with conductivity measurement and filling tap (test operation)

Figure 9: Demineralizer AFT VE-Station 100 Mono (final solution)

The sample preparation in the laboratory was also critically examined. It has been found out that a filtration of the samples from the washing machine is necessary because larger textile fibers in the sample can clog the flowthrough cuvette of the spectrofluorometer. Furthermore, smaller, slightly fluorescent fiber constituents can increase the blank value. Therefore, each sample was transferred through disposable filter paper (Macherey-Nagel MN 615) to another vial prior to analysis. However, preliminary investigations with comparative samples showed that even the filter paper itself flushes out slightly fluorescent substances which cause the blank value to increase indefinitely. For this reason, the filtration was switched to stainless steel sieves (Retsch test sieve) with a mesh size of 250 µm. These must be carefully cleaned with distilled water after each sample, but the uncertainty resulting from the filter paper can thus be reliably and permanently avoided.

Figure 10: Setup for filtration with filter paper

Figure 11: Setup for filtration with stainless steel sieve (250 µm mesh size)

2.3.2 Dosimeters

For the experiments in the current project similar dosimeters are used as in the EasyFlow project. Furthermore, long underwear and a protective overall are used as a body dosimeter. In addition, protective gloves are still worn as outer hand dosimeter. The cotton gloves as inner dosimeters have been surrogated. Instead, disposable laboratory gloves are worn directly under the protective gloves and evaluated as an inner hand dosimeter. As a head dosimeter, the protective visor continues to be worn in front of the face. The filter paper on the neck is waived because the contamination risk of this body part showed to be extremely low. In various preliminary tests, all dosimeters were thoroughly tested for their suitability and, if necessary, sought for better alternatives.

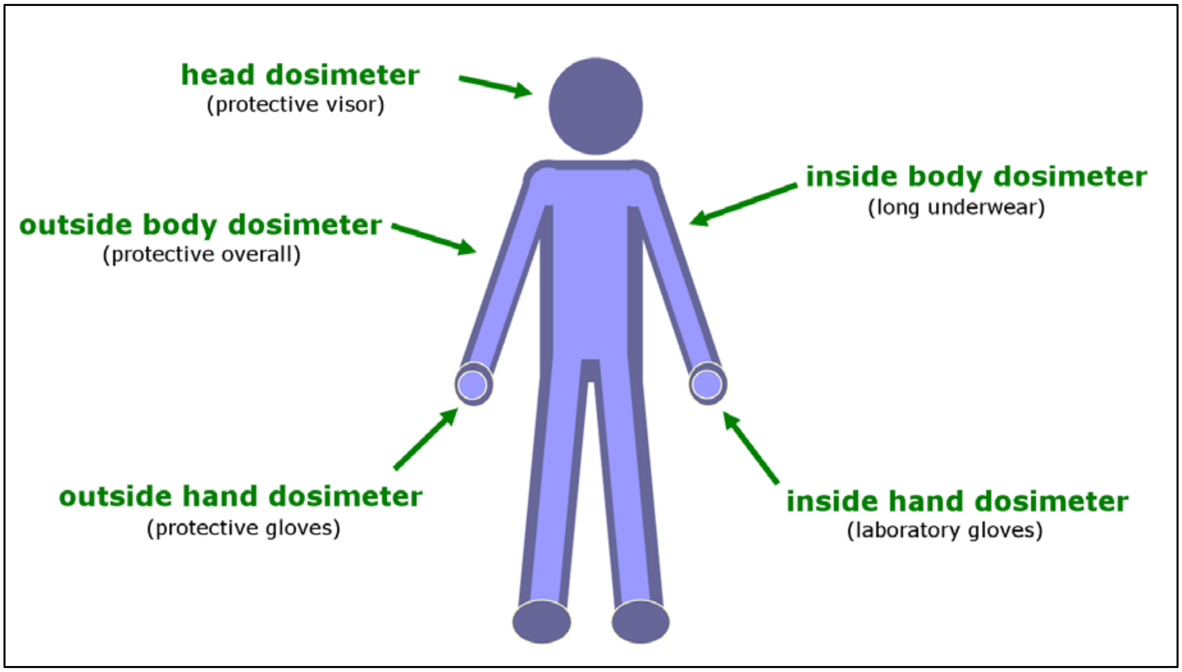

Figure 12: Overview of the dosimeters used in the CTS project

First, various protective overalls were examined. The white one-piece overall (STONEKIT overall, white) from the EasyFlow project was compared with a one-piece brown overall (STONEKIT overall, brown) and a two-piece white overall (Wolfgang Mauser GmbH jacket and trousers, white). It could be proved that the new two-piece overall of 65% cotton and 35% polyester had a significantly lower blank level than the two comparative overalls. The calculated recovery rates were also satisfactory. The examination also showed that all overalls in new condition have a relatively high blank value, which drops steeply over the first washes and converges to a limit. It is therefore necessary to pre-wash the dosimeters several times before use.

New underwear made of 100% cotton (Pfeilring UNDERWARE, white) was available for the CTS project. Preliminary tests with already pre-washed dosimeters showed sufficiently low blank values and good recovery rates. Further investigations showed, however, that the blank value was again extremely high on some test days. Through specific tests, it was found that the material releases fine fiber particles after drying and after movement, which cause the blank value to increase by an unforeseeable amount. Since drying and wearing are unavoidable, a test underwear made of 100% polyester (Engelbert Strauß functional longsleeve and long pants base light, white) was procured. The blank values of this material were extremely low and stable. Also drying or movement did not lead to changes. In recovery attempts, it quickly became apparent that the tracer is not absorbed by the material as in cotton. Instead, the drop remains permanently on the material surface or rolls off completely from the material. Since the underwear as an internal dosimeter is intended to simulate the skin load of the operator and absorb the respective amount of tracer, this material also had to be classified as unsuitable.

Figure 13: Drop of the tracer, which rests outside of the polyester fabric and rolls off

Figure 14: In the case of contamination of the overall jacket, no tracer is absorbed by the underlying polyester underwear, and instead the drop only spreads within the jacket fabric

Since effects such as changes in the blank value due to drying or lack of absorption capacity had not been noticed in the predecessor project, the stock from the EasyFlow project was used again. It turned out that the underwear made of 95% cotton and 5% spandex (Engelbert Strauss cotton stretch longsleeve and long pants, white) is indeed a very good compromise. Although an increase in the measured values after drying is measurable, the value of the increase over several preliminary experiments was at a level which was still clearly below the detection limit. The achievable recovery rates were also satisfactory with lower contamination levels.

In addition to new overalls and underwear, new nitrile gloves (MAPA Stansolv AK22) were also available for the CTS project. These were also examined for their suitability with regard to stable blank values and the level of recovery rates. While the blank values remained stable at a low level, it was found that the calculated recovery rates were extremely low. At the same time, it could be observed that the samples with pyranin-contaminated gloves had a clearly recognizable green color of the water. The comparison with a control dilution of the tracer liquid, which theoretically contained the same pyranin concentration and was barely discernible, showed that the green color is not only caused by the pyranin (see Figure 15). Since the blank samples without pyranin were also not discolored, it is assumed that ingredients on the outside of the gloves react with the pyranin, which degrades it and turns the water green. The cotton velour-coated inner side of the gloves could be ruled out by further experiments with gloves turned "on the left side" as a source of reaction.

Figure 15: Green color of the water in the recovery experiment (three glasses on the right) compared to the blanks (on the left) and for dilution with the same concentration (large glass in the background)

Since the existing protective gloves gave unsatisfactory results, experiments were carried out with remaining stock from the predecessor project (KCL Camatril 730), in which the problem had not occurred. The suitability of these gloves could again be fully confirmed.

As in the predecessor project, the gloves are placed individually in closed PET containers of 500 ml capacity after the tests. Before analysis, each container is filled with 200 ml of distilled water and swirled for 20 minutes on a laboratory shaker at 75 min^{-1}. After about 10 minutes, each container is rotated 180°, so that both sides of the glove are sufficiently wetted with water and rinsed. The same procedure is applied to the laboratory gloves.

Figure 16: Protective gloves and laboratory gloves in PET containers on the laboratory shaker

The laboratory gloves were disposable gloves. First, latex gloves (semper guard latex powder free) were tested. The blank value of these gloves are very low and the calculated recovery rates were slightly lower than the recovery rates of the protection gloves, but still good at an average of 92.2%. They were therefore used for the first filling tests.

In order to clarify whether nitrile laboratory gloves (semper guard nitrile powder free) are suitable, a comparative test was carried out parallel to the first filling experiments. The test results showed, on the one hand, that the blank values and, in connection therewith, the detection and quantification limits of both materials are at a comparably low level. On the other hand, the nitrile gloves achieved higher recovery rates, while the recovery rates of the latex gloves were lower compared to the first test results. Striking in these experiments was that the samples of latex gloves with pyranin concentration turned slightly greenish. This effect is reminiscent, albeit less pronounced, of the problem with the protective gloves. For this reason, the latex gloves in the filling experiments were replaced by nitrile gloves from that moment on.

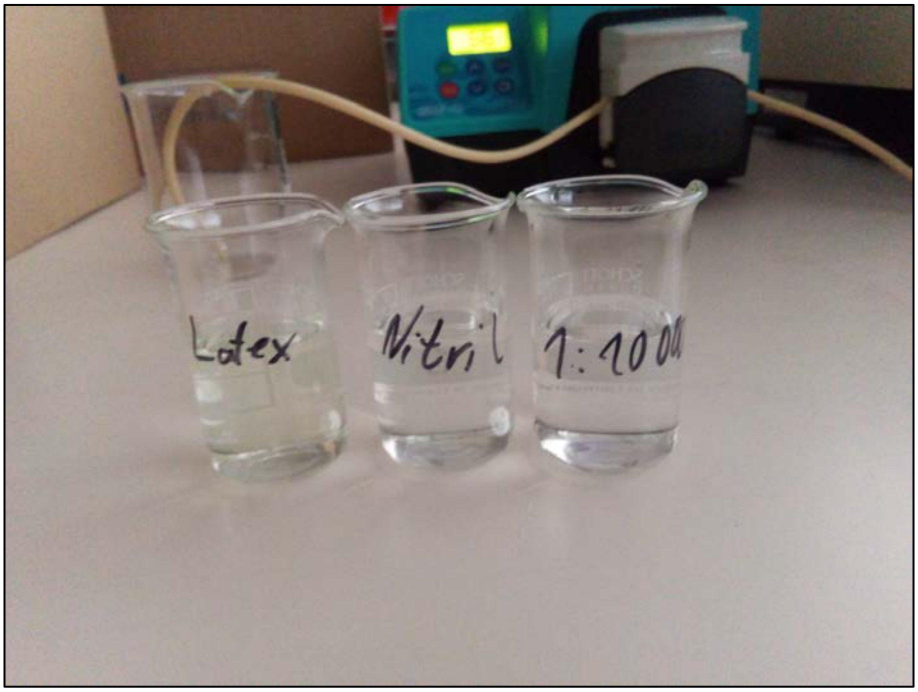

Figure 17: A slight discoloration is also found in the latex laboratory gloves (left) compared to the nitrile laboratory gloves (center) and the control dilution with the same concentration (right)

The protective visor (ekastu k1 plus) from the EasyFlow project was still used unchanged. It is a divisible combination of a visor holder and a transparent visor shield made of polycarbonate (PC). The visor shield is also elastic and can be easily connected or disconnected via three pushbuttons with the visor holder. This fact favors the rinsing of the tracer, since the removed visor shield can be rinsed over a collecting container with 400 ml of distilled water. This process is easy to handle manually with a squirt bottle. The blank value is almost at the level of distilled water and the recovery rates determined in the preliminary test are very high. A disadvantage of the material is that only very small drops adhere to the surface. Large drops would roll off due to lack of adhesion.

Table 1: Recovery rates from preliminary experiments in the laboratory

Dosimeter	**Material**	**Average recovery rate (reference quantity)**
Visor	Polycarbonate	101.0 % (20 µl)
Protective gloves	Nitrile with cotton inner velour	99.6 % (20 µl)
Laboratory gloves	Nitrile	91.2 % (20 µl)
Overalls	65 % cotton / 35 % polyester	102.0 % (25 µl)
Underwear	95 % cotton / 5 % polyester	93.1 % (25 µl)

Figure 18: Outer dosimeters (protective overall, protective gloves and protective visor)

II.3.3 Calculation of the dosimeter characteristics

For the exact calculation of the tracer concentration, it is important to determine the blank value. This is different for each type of dosimeter and has to be deducted from the corresponding FI measured values, since it is a self-fluorescence of the material and not a tracer contamination. The blank value is determined as an average from several measurements with contamination-free dosimeters. With a sufficient number of blank value samples, the detection and quantification limit can also be determined directly from this. The following calculation formula apply for the blank value B, the limit of detection LOD and the limit of quantification LOQ:

$$B = \frac{\sum_{i=1}^{n} x_i}{n}$$

$$LOD = B + 3 \cdot s$$

$$LOQ = B + 9 \cdot s$$

with the empirical standard deviation s:

$$s = \sqrt{\frac{\sum_{i=1}^{n} (x_i - B)^2}{n-1}}$$

B = Blank value

x_i = FI measurements from contamination-free dosimeters

n = number of measurements

s = Empirical standard deviation

LOD = Limit of detection

LOQ = Limit of quantification

The laboratory and protection gloves are only used once. Therefore, the blank values do not change over time because new gloves are available for each test. The protective visor made of PC is used multiple times. However, it is rinsed off only with water and a measureable dissolution of material particles from the visor is not possible. Therefore, no change in the blank value with time is to be expected here either.

Somewhat more complicated is the determination of the characteristics of overalls and underwear. Fiber particles and additives which affect the measured fluorescence intensity are rinsed from the textiles in the washing machine with time. For this reason, all new textile dosimeters were prewashed eight to ten times in advance in a washing machine with normal tap water and a longer washing program in order to remove a large part of the foreign substances in advance.

Only then, the washing of the dosimeters started with demineralized water and samples were taken and analyzed. It was found that the blank value was significantly reduced with the first three to five washings before a stable value was reached. Due to this variability, the blank value must be followed up during the trials, if the dosimeters are to be used several times. To do this, the textile dosimeters are washed three times after each test. While the samples of the first two washes are used to determine the tracer content (it is possible that a small amount of pyranin may still be present in the second washing), the third washing serves as a blank sample. After completion of all tests, the blank value can be calculated from the measured values. It is assumed that the water volume of all blank samples is constant. The slight variation in water volume that occurs in reality is not taken into account in the calculation, as the analysis of the data has shown that other factors have a much greater influence on the blank values.

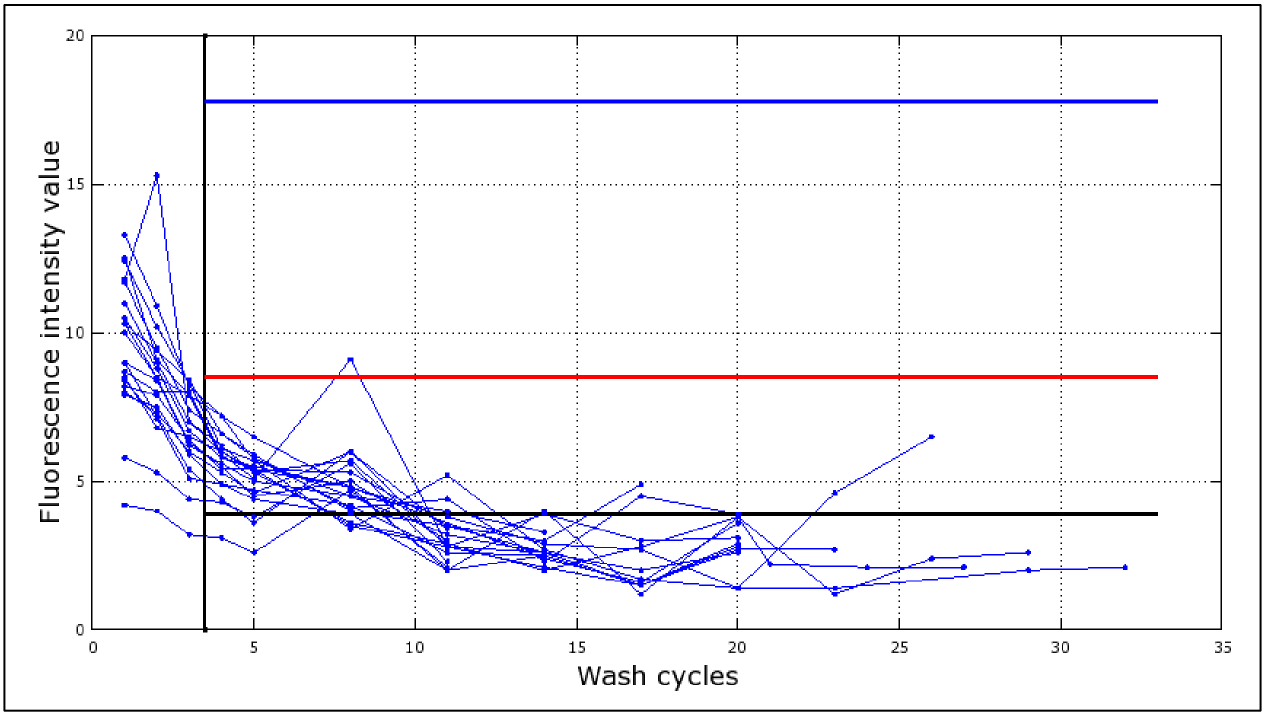

Figure 19: Blank value history of the new overalls over the number of washes and average values of the calculated blank values as well as the detection and quantification limits

As a result of the steep drop in blank value at the beginning, the samples from the first three pre-washings were always excluded from the blank value calculation. All other blank value samples are included both in the calculation of the blank value and in the calculation of the detection and quantification limit. Since blank samples from each individual dosimeter are available in this procedure, it is possible to calculate the characteristic values for each individual dosimeter separately in order to be able to detect differences between individual clothes. In order to achieve a high accuracy this procedure was used for the evaluation of the overalls and underwear from the trials. The diagram in Figure 19 shows the actually measured blank value curves of all overalls. In addition, the average value of all individually calculated blank values and the average of all individually calculated detection and quantification limits are plotted. The actual characteristics thus spread in a narrow band around the averages shown. A uniform blank value for the entirety of all overalls or underwear is only meaningful, if it is ensured that all dosimeters of a type are preconditioned equally as well.

2.4 Trial program

The trial program in the CTS project is divided into two parts. In the first part of the experiments, seven different operators individually carry out filling experiments on the induction hopper without CTS with different canister sizes. This is to identify a "worst case" scenario for the canister size leading to highest operator contamination. In the second part of the experiments, only three different operators carry out filling experiments. In this part, the four filling variants from the EasyFlow project are continued. The "worst case" canister identified in the first part of the experiments is used for these tests. The trial variants are performed by each operator in five repetitions.

2.4.1 Experimental setup

The experimental device used is a Rau D2 tractor-mounted field crop sprayer with a capacity of 700 liters. An easyFlow tank adapter was installed on the cover of the induction hopper as well as on the tank cap of the dome shaft in accordance with the manufacturer's operating instructions. To fill the field crop sprayer via the induction hopper, the pump was driven by an electric motor with reduction gear and cardan shaft at a speed of 540 min^{-1}. With a rotary valve, the injector of the induction hopper can be put into operation. The canister rinsing nozzle was used to rinse the canisters. The circulation rinsing pipe squirted fine droplets and was therefore not used in the experiments, since the exposure potential during cleaning is highly dependent on the particular design of the induction hopper and the experimental results might have been distorted. The upper filling edge of the induction hopper was at a height of 1.0 m and the upper filling edge at the dome shaft was at 1.55 m. For safe filling of the field crop sprayer on the dome shaft therefore a commercial stepladder was used.

Figure 20: Induction hopper of the field crop sprayer

Figure 21: Dome shaft with easyFlow tank adapter

Figure 22: Experimental setup of the field crop sprayer with drive motor

The mixing and filling of the pyranin solution into the canisters took place locally in advance to the filling experiments. The amount per mixing process was chosen as needed. As a mixing tank, a rain barrel with 310 liters nominal volume and drain tap was used. The amount of water was metered by means of a volume meter (Sotera Systems, 850 Digital Meter) on the hose line. The powdery pyranin was weighed in a bucket, added and mixed with a stirrer. After the solution was completely homogenized, a sample was taken from the mixing tank. Subsequently, the filling into the canisters was done using the drain tap in the lower part of the mixing tank. To achieve the nominal filling quantity as accurately as possible, a balance was used (Kern ITB 35K1IP). Any contaminations of the canister outside were thoroughly removed with a paper towel and water.

Figure 23: Filling of the canisters from a common mixing tank

The sealing of the filled canisters was also carried out locally by means of a portable sealing device (enercon Super Seal Junior Sealer ML0104-701-03), which heats the sealing foil contained in the sealing cap by induction and welds it to the canister. The sealed canisters were then packed on a pallet and stored under absence of incidence of light. While only two fillings of the canisters had to be carried out for the entire period of the first part of the experiments, from the second part onwards, due to a larger quantity required and in favor of shorter storage times, a separate filling of the canisters was carried out before each trial day. The taken mixing tank samples were each used to calibrate the associated measurements.

Figure 24: Sealing device with canisters and caps

Figure 25: View of a 5 liter canister after sealing

The Good Agricultural Practice defines that spraying of pesticides hat to be avoided at wind speeds above 5 m/s, at temperatures above 25 °C or below 30% air humidity. (BMELV 2010.) In contrast to the previous project, the filling tests were not carried out outside but in a closed test hall in the basement of the Institute of Application Techniques in Plant Protection. This makes it possible to ensure that the tests can be carried out regardless of the weather conditions and under comparable and controlled conditions, which fulfill the requirements of Good Agricultural Practice. The temperature and relative humidity were measured and documented on each trial day using a hand-held meter (testo 625) (Figure 26). The measurement of wind speed was waived due to the testing location being inside.

Figure 26: Hand-held meter (testo 625) for measuring temperature and humidity

2.4.2 Experimental procedure

On each trial day, several filling experiments can be carried out as long as it is ensured that they do not interfere with each other due to the contamination. In each of these individual experiments, one operator dressed with inner and outer dosimeters performs the predefined filling cycle. Then the dosimeters are packed and a new filling experiment can be carried out. The first experiment on each day was a control experiment to determine the recovery of textile dosimeters under trial conditions (field recovery). For this purpose, a contamination-free filling experiment was carried out with clear water instead of the pyranin solution. The overall was then contaminated with a pipette from the outside with 50 µl pyranin solution (split on jacket and trousers).

The filling cycle was defined in advance and in this project always includes the filling of four canisters. In order to take account of the ability of modern CTS that they also allow partial emptying, the first canister is only partially emptied, placed back and completely emptied at the end. The second, third and fourth canister, on the other hand, are immediately completely emptied.

Figure 27: Carrying out a filling test at the induction hopper without CTS

The filling process on each canister begins with unscrewing the cap. Thereafter, the sealing foil is cut with the back of the cap and the contents emptied directly into the sprayer tank or the induction hopper. Finally, each canister is rinsed and closed again with its cap. When using the CTS, the manual opening of the sealing foil is not necessary and the easyFlow system is used instead, following the manufacturer's operating instructions.

Depending on the mounting position, it is important that additional units needed for the process are switched on (e.g. injector nozzle and canister rinsing nozzle on the induction hopper). This may perhaps be relevant to contamination if the operator comes into contact with contaminated device components during the filling process. Thus, in the experiments on the induction hopper, switching on the injector at the beginning and switching off at the end of the filling cycle were part of the filling experiment. Similarly, in the experiments on the dome shaft without CTS the tank cap was opened at the beginning and closed again at the end.

In the first part of the experiments, each of the seven operators filled one canister of each size (1 liter, 5 liters and 10 liters) via the induction hopper. No CTS was used here. Thus, the experiments take into account the current state of the art. It should be determined if the different canister sizes differ in their contamination potential, in order to find out a “worst case” scenario.

In the second part of the experiments, the previously determined "worst case" canister was used for different filling variants. Now, filling experiments were carried out on the induction hopper and dome shaft. For both positions, both the conventional filling process without CTS and the filling process with CTS were carried out. The four filling variants were run by three different operators in five repetitions.

Figure 28: Induction hopper without CTS

Figure 29: Dome shaft without CTS

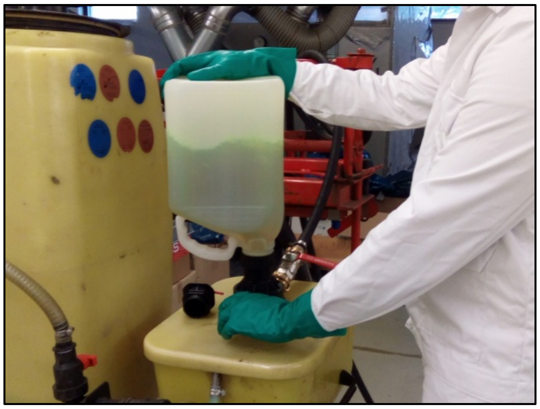

Figure 30: Induction hopper with CTS

Figure 31: Dome shaft with CTS

2.4.3 Operators

The operators who carry out the filling experiments are of particular importance, since they differ considerably in terms of their body size and strength as well as their level of experience and their habits. To perform the first part of the experiments, seven operators were selected to get a picture of the individual differences. For the second part of the experiment, the number of operators was reduced to three persons where one person was not involved in the first part of the experiments. All test persons were JKI employees who had at least the certificate of competence for use in plant protection. Individuals were selected to be different in body size, physical strength, gender and application experience. This ensures that the validity of the test results is not limited to a specific group of operators. For the different body sizes the overalls and laboratory gloves were available in different sizes. The underwear made of elastic material and the pesticide gloves were only available in one size, but fit all operators. The headgear of the visors could be adapted to the personal requirements of each individual.

All operators were instructed on how to put the body dosimeters on and off to avoid cross-contamination between the dosimeters and to package them properly. Assistants helped them removing the visor, protective gloves and laboratory gloves. The canister order of the filling cycle was also explained in the instruction. All operators were required to behave as they are used to in the filling process (for example, from their own farming). They were instructed in the operation of the field crop sprayer and in particular in the operation of the easyFlow system in accordance with the manufacturer’s operating instructions. None of the test persons were experienced in using CTS, all of them used it for the first time during the trials.

3 Trial results

3.1 First part of the experiments

3.1.1 Results

The dosimeters of the individual experiments were analyzed separately and the exposures were calculated taking into account the respective detection and quantification limit. The evaluation of the experiments was done separately by canister size using the 75th percentile. The Excel function QUANTIL (_: _; 0.75) used for this interpolates the result if necessary. Average values and standard deviations were also calculated as additional information. The results are presented here in the form of a tracer volume (volume of solution) with the starting concentration contained in the canister and not as a tracer mass to facilitate interpretation with respect to a liquid PPP. A conversion into mass data is possible at any time by multiplication with the initial concentration of 5 g/l.

Visor

During the first part of the experiment, it was only in one case possible to detect an excess of the detection limit on the visors. This measured value, which occurred during a filling process with the 5 liter canister and lies between the detection and quantification limits of the visors, does not affect the overall evaluation using the 75th percentile. As the detection and quantification limits are very low, the average exposure level for this case is also extremely low.

Protective gloves

For the protective gloves, all measured values exceeded the detection limit. Only in one case when using the 1 liter canister, the measured value was between the detection and quantification limit. This provides sufficiently meaningful exposure data for this dosimeter. Evaluation by the 75th percentile shows higher exposure for larger canister volumes. In particular, with the 10 liter canister, the exposure amount of 21.9 µl is more than twice as high as that of the 1 liter canister with 10.4 µl. The average values lie below the 75th percentile and confirm this tendency accordingly.

Laboratory gloves

In the first part of the experiment, it was never possible to detect an excess of the detection limit on the laboratory gloves, although the detection limit is relatively low.

Overalls

Exposure of the overalls was found only on the larger 5 and 10 liter canisters. There are two findings in each case, with one measured value above the limit of quantification and one measured value between the detection limit and the limit of quantification. Exposure levels are highest for the 10 liter canister, which also indicates higher exposure for larger canister volumes. However, the majority of the measured values were below the detection limit and therefore cannot be determined quantitatively. In four out of seven cases an exposure could not be detected at all.

Underwear

In some cases, underwear measurements exceeded the detection limit. An exceeding of the limit of quantification was not found. Calculations determined an exposure of up to 1.0 µl using the 1 liter canister. However, this exposure occured only in a single case at the same time with an exposure of the associated overall.

Total exposure

The sum of the exposures is decisive for the assessment of the exposure risk. For this, the results of the single dosimeters for different canister sizes were added. The total exposure is also at a much higher level for larger canister volumes. Also, the total exposure amount when using the 10 liter canister with 25.1 µl is more than twice as large as when using the 1 liter canister with 11.4 µl. With regard to the distribution to different dosimeters, it is noticeable that the Protective gloves account for by far the largest share of the total exposure. The exposure of the overalls accounts for only a smaller proportion and is only relevant for the larger canisters. The exposure of underwear makes only a very small and approximately constant proportion of the total exposure.

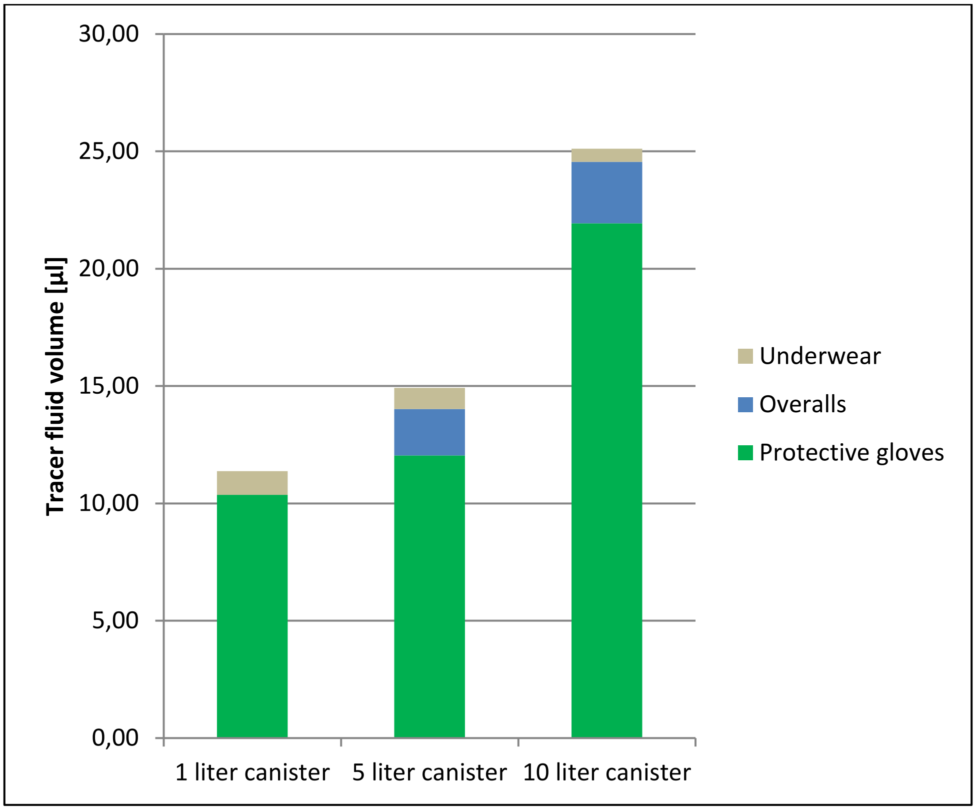

Figure 32: Total exposure as the sum of the 75th percentile of the exposed dosimeters for different canister sizes from the first part of the experiments

Detection limits and quantification limits

The amount of the detection and quantification limits differs in part significantly for different types of dosimeter. Thus, the limits for the textile dosimeter overall and underwear are significantly higher than the values for visor and gloves. The reasons for this are on the one hand in the much larger amount of water, which requires a higher measurement sensitivity, and on the other hand in the higher input of foreign particles from the textile structure. As previously mentioned, the values for overalls and underwear are calculated individually. Only the average values of all dosimeters of a type are shown in Table 2.

Table 2: Detection and quantification limits in the first part of the experiments

Dosimeter	Detection limit (µl)	Quantification limit (µl)
Visor	0.1	0.3
Protective gloves	0.34	1.01
Laboratory gloves	0.08	0.24
Overalls	2.45*	7.34*
Underwear	0.99*	2.97*

*Average

The recovery rates of the textile dosimeters were checked once a day under experimental conditions. Thus, seven values were determined for the first part of the experiments. After carrying out a control filling cycle using clear water canisters, the overalls of the operators were contaminated externally in two places with the tracer. An amount of 50 µl of the tracer solution was applied via pipette. In several cases - due to the direct application technique via pipette - the underwear was slightly contaminated, too. Therefore, the recovery rate was always established based on the exposure of the overall and underwear. The average recovery was 85.2 % with a standard deviation of 8.9 %. The lowest value was 72.9 %, while the highest value was 94.9 %. In addition, the protective gloves were checked in the first part of the test series on four days parallel to the general control. Again, 50 µl of the tracer solution was applied to both gloves. The average recovery of the gloves was 99.9 % with a standard deviation of 8.2 %.

3.1.2 Observations

The first part of the experiments was carried out by seven different operators. Therefore, many minor differences between the operators were observed. The decisive difference has been the handling of the sealing foil. This sealing foil was partially cut open with the cutting edge on the back of the cap and the canister thus fully opened. Only one out of seven operators started directly with the filling process afterwards. Two operators pressed the sealing foil with their finger into the inside of the canister, whereby the foil was often flushed outwards during the filling process by the liquid flow. With four operators, the majority of the test persons have completely demolished the sealing foil by hand and disposed it separately so that the sealing foil cannot get into the field crop sprayer during the filling process. In particular, when tearing off the sealing foil, the gloves usually already come into direct contact with the tracer solution.

With regard to the different canister sizes, it has been noticed that the handling of small canisters is considerably easier than with larger ones. 1 liter canisters could often be held in the lower part of the canister with only one hand, while their opening was directly within the induction hopper. The larger canisters had to be hold by the handle in the upper part and were held with their opening rather above the induction hopper as inside. As a result, the open flow path is usually longer for larger canisters and the liquid has a higher energy, which can lead to splashes when hitting the wall of the induction hopper.

3.1.3 Conclusions

The results of the first part of the experiments show that the potential operator exposure is highest for the 10 liter canisters. Several reasons seem to play a role here. On the one hand, with the 10 liter canister, the absolute amount of liquid is the highest. This also increases the likelihood that a larger part of the liquid quantity will reach the operator's body. Furthermore, the ratio between the canister volume and the cross section of the canister opening is highest. This leads to higher pressure fluctuations during the outflow of the liquid or the associated inflow of air. As a result, the flow rate is harder to control and the risk of splashing increases. In addition, the handling of the canister is generally more difficult due to the high weight and the voluminous design. Also, the open flow path is longer because of the size. The 10 liter canister is thus used for all experiments of the second part of experiment to depict the "worst case" scenario.

A closer look at the results reveals that protective gloves account for the largest percentage of operator exposure. This is consistent with the results from the EasyFlow project, although the amount of exposure was slightly larger. The reason for the greater percentage of the protective gloves is that the hands are very close to the canister opening during the filling process and, when handling the canister, partly come into direct contact with product on the sealing foil or on the outside of the canister. Since the Protective gloves are completely leak-proofed, no exposure of the underlying laboratory gloves could be detected. The overalls also contribute to the total exposure of the two larger canisters. It can be noticed that in many cases an excess of the detection limit was determined on the underwear, so that it also adds to the total exposure for all canister sizes. Since the underwear was worn completely under the overall, an exposure of the underwear would have physically preceded any exposure of the overall. The fact that the exposure values of overalls and underwear match only in one case for the 10 liter canister suggests that this is not always a real exposure of the underwear. Rather, material effects caused by drying the textiles between the experiments seem to cause the detection limit to be exceeded. Since the visor on the operator's head is usually worn automatically with a large distance to the work area, this dosimeter is hardly endangered to exposure.

3.2 Second part of the experiments

3.2.1 Results

In the second part of the experiments, the dosimeters were also analyzed separately and the exposure was calculated considering the respective detection and quantification limit. Here the evaluation of all experiments was done separately for the four filling variants and again using the 75th percentile (Excel-Funktion QUANTIL(_:_;0.75)). Averages and standard deviations were also calculated as additional information. The form of presentation as a tracer volume was maintained.

Visor

In two cases measured values between the detection limit and the quantification limit occurred at the dome shaft with CTS. These do not affect the overall evaluation using the 75th percentile. As the detection and quantification limit is very low, the average exposure level is also extremely low.

Protective gloves

In the case of protective gloves, a measured value above the limit of quantification was determined in all cases both at the induction hopper without CTS and at the dome shaft without CTS. It is noticeable that the amount of the 75th percentile at the dome shaft is about twice as large with 28.7 µl as at the induction hopper with 14.2 µl. When using the CTS, only very few values are above the limit of quantification. In many cases, however, the detection limit was exceeded. By using the CTS exposure levels have been significantly reduced by 95.2 % at the induction hopper and by 97.6 % at the dome shaft utilization. The average values are below the 75th percentile and clearly confirm the reduction in hand exposure.

Laboratory gloves

Exceeding the limit of detection of laboratory gloves was found in only one case at the induction hopper without CTS. This does not affect the evaluation using the 75th percentile. Also, the average value is extremely low.

Overalls

Exposures of the overalls occurred in all four filling variants. In most cases, however, the detection limits were not exceeded here either. After evaluation by the 75th percentile exposure could only be detected in two filling variants. In the variant at the induction hopper without CTS this was 9.2 µl and at the dome shaft without CTS 2.7 µl could be found. The average values are above the 75th percentile in all filling variants.

Underwear

In some cases, underwear measurements exceeded the detection limit. Only in the variant at the dome shaft without CTS no exceeding of the detection limit was found. The quantification limits were not exceeded. After evaluation by the 75th percentile, an exposure of 2.0 µl was found in the variant at the induction hopper without CTS. In many cases the calculated exposures of the underwear do not coincide with exposures of the corresponding overalls.

Total exposure

The total exposure during the filling process at the dome shaft without CTS is with 28.7 µl slightly higher than the value determined in the first part of the experiments with the 10 liter canister, while the value during the filling process at the induction hopper is slightly lower with 23.4 µl. As in the first part of the experiments, the protective gloves have by far the largest proportion of the total quantity in conventional filling. A considerable contribution to the total exposure arises at the induction hopper without CTS also by the overalls. At the dome shaft with CTS, the exposure amount of the overalls is even more significant than that of the protective gloves. A reduction of the total quantity by the CTS is evident in each case. The calculated total reduction using CTS sums up to 88.8 % at the induction hopper and 88.2 % at the dome shaft.

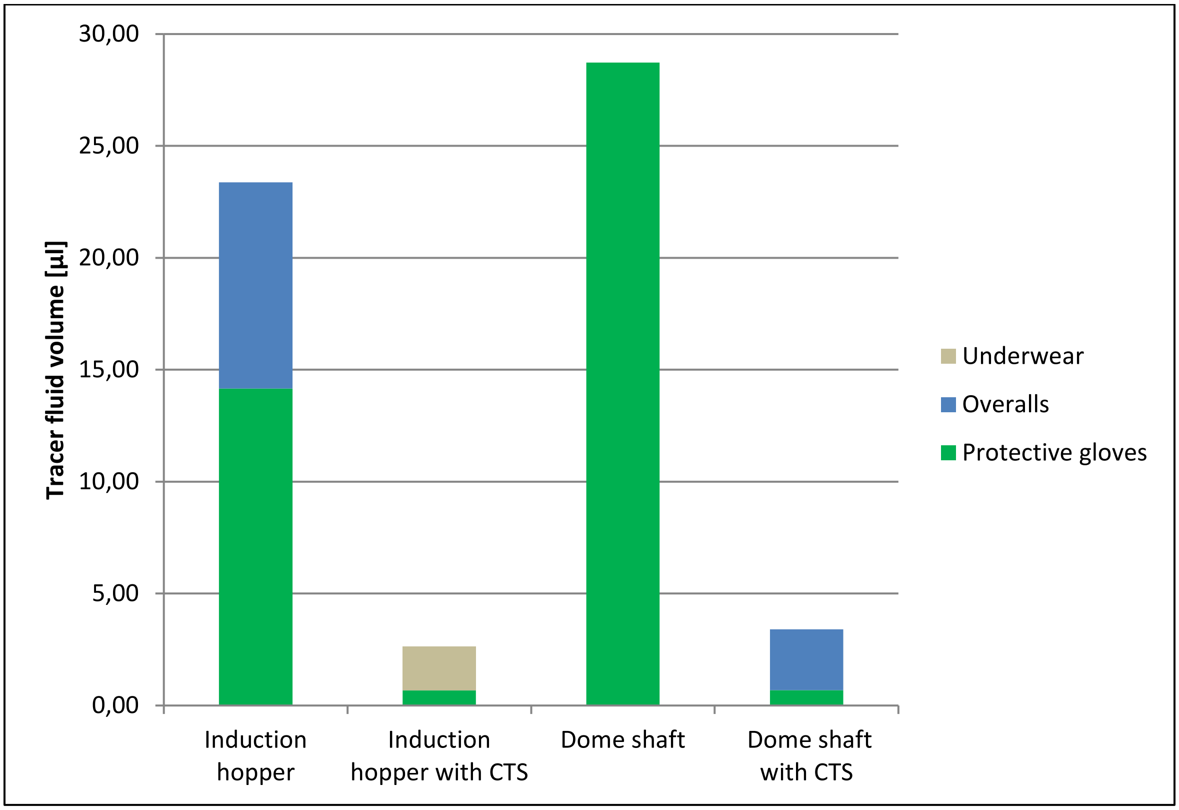

Figure 33: Total exposure as the sum of the 75th percentile of the exposed dosimeters for the different filling variants from the second part of the experiments

Detection and quantification limits

The detection and quantification limits for visor, protective gloves and laboratory gloves were retained in the second part of the experiment. In contrast, the limits for overalls and underwear have been recalculated with the now available larger amount of dates. Again, in Table 3 average values of all dosimeters of a type are shown.

Table 3: Detection and quantification limits in the second part of the experiments

Dosimeter	**Detection limit (µl)**	**Quantification limit (µl)**
Visor	0.1	0.3
Protective gloves	0.34	1.01
Laboratory gloves	0.08	0.24
Overalls	3.26*	9.78*
Underwear	1.7*	5.1*

*Average

Field Recovery

For the second part of the experiments, twelve values for the recovery rate of textile dosimeters were determined under test conditions. Again, this is the sum of the exposures of overall and underwear. The average recovery was 82.1 % with a standard deviation of 25.3 %. The lowest value of 46.7 % was less than half, while the highest value of 131.8 % was clearly above 100 %. This variation of the recovery rate is relatively strong in comparison with the first part of the experiments.

3.2.2 Observations

Also in the second part of the experiments with only three operators, the handling of the sealing foil in the variants without CTS was a decisive source of exposure. One operator pushed the sealing foil in with his fingers. The other two operators removed the sealing foil completely.

At the beginning, the handling of the CTS was sometimes difficult for the inexperienced operators, since various non-intuitive sub-steps must be carried out in the correct order. However, it is unlikely that this initial uncertainty could have influenced the measured results. Only in one case at the dome shaft, the canister adapter could not be stretched properly after rinsing and the respective operator therefore handled the already removed canister adapter for an unusually long time in different grip positions. Since this was not a use within the scope of the operating instructions, the corresponding measured value was excluded from the evaluation and is therefore not included in the results.

Since the duration for the washing procedure of the emptied canister is not specified in the operating instructions, it also came to differences between the operators. The canister was in all cases rotated at least 360 ° around its own vertical axis during the rinsing process in order to wet the entire inner surface with rinsing water. Depending on the operator and situation, the rotational speed varied. One operator rotated the canisters several times around its own axis. In all cases, the canisters were visually clean after washing.

An important observation in all experiments with the CTS was that even after the rinsing process residual amounts of the tracer solution were still visible on the contact surfaces and on the inside of the canister adapter (see Figure 34 and Figure 35). The liquid residue on the contact surfaces can run down when unscrewing the canister adapter and lead to a subsequent exposure of the operator. In fact, in some experiments it could be observed that droplets dripped from the contact surface onto the protective gloves during unscrewing. After unscrewing, the inside of the canister adapter is also open. At this point it is possible that residual quantities flow out and lead to a subsequent exposure of the operator, e.g. on the overall. This could be observed at least in one case at the dome shaft with CTS. However, no quantitative statement can be made about the level of concentration of the residual amount, since pyranin is still clearly visible even in high dilution.

Figure 34: Contact surfaces of the canister adapter after the filling cycle

Figure 35: Inside of the canister adapter after the filling cycle

3.2.3 Conclusions

The results show clearly that the operator exposure is reduced by the CTS. With regard to the protective gloves, the results from the second part of the experiments confirm the data already obtained in the EasyFlow project. In addition, no relevant exposure of the inner hand dosimeter or head dosimeter was found. With regard to the overalls, the detection limit and the quantification limit were each reduced by more than half compared to the predecessor project. Accordingly lower exposures could be measured. Nevertheless, it was found that even under these enhanced conditions, in many cases, the detection limit is not exceeded. The number of detectable exposure events is therefore relatively low, although the absolute level of exposure may be high in some cases. The detection limits of the underwear were often exceeded. In this project, the quantification limit was only exceeded for underwear in a few field recovery experiments where the tracer solution was manually applied directly to the overalls. The tracer amounts determined in the exposure tests are therefore relatively low compared to overalls and protective gloves. In addition, the measured data of all individual washings show that exceeding the detection limit could at least partly be due to material effects caused by the drying of the textile material. The fact that the underwear exposure often does not match the overall exposure supports the suspicion that there has not always been a real exposure.

The variation of the measured results is relatively high for the conventional filling process. By comparison, measurements using the CTS spread much less, as the level of exposure for CTS is lower. Again, however, a sufficiently high number of repetitions with different operators is advisable in order to minimize the influence of situational or operator-related differences on the results. This applies in particular to the measurements of the overalls, since detectable exposure events are rare.

The fact that the exposure in the variants at the dome shaft is higher than in the comparison variants at the induction hopper probably depends on the fact that due to the height the operators had to use a stepladder to reach the tank opening. This leads to a greater movement uncertainty of the operator when handling the canister or the filling system. In addition, the risk that the operator is contaminated by contact with the surface of the field crop sprayer or the ladder is higher at the dome shaft compared to the induction hopper.

3.3 Examination of residual quantities at the canister adapter

In order to be able to classify the quantity of liquid adhering to the canister adapters after the filling process, an additional trial was carried out. Therefore the tank adapters were carefully unscrewed immediately after the filling process and packed directly into a sealable container. The canister was then filled with distilled water and swirled on the laboratory shaker to rinse off the pyranin completely. The pyranin concentration was then determined by fluorometry and thus the residual amount was calculated.

To carry out the experiments, a clean canister adapter was used to fill a 10 liter canister at the dome shaft of the field crop sprayer (Rau D2). The canister was completely emptied at once. Then the canister was rinsed clean according to the operating instructions. The clean canister was unlocked, removed smoothly from the tank adapter and slowly rotated 180°, so that the canister adapter is back up. Then it was unscrewed from the canister and packed directly into a provided, labeled container. The particularly careful movements should ensure that as far as possible the entire amount of liquid present on the adapter is collected. Four repetitions were performed. In two cases the canister adapter was stretched in locked position on the field crop sprayer and in the other two cases, the adapter was left unstretched.

For analysis, each container was filled with 2 liters of distilled water so that the canister adapter was completely wetted with water inside and out. Thereafter, all containers were placed on the laboratory shaker for 20 minutes at 70 min^{-1}. Based on a stock solution, which was also used for calibration, the measuring range of the spectrofluorometer was set. Sampling from the individual canisters before the filling test was technically not possible because of the closed sealing. Therefore, the results were first calculated based on the calibration with the stock solution. The concentration of the canister solution was calculated from the available data on pyranin mass and water quantity per canister. Correction factors were used to correct the volume results for the small concentration differences.

The individual measurement results between the four repetitions showed great differences. However, quantities in the milliliter range were found in each case. The average was 5.5 ml with a standard deviation of 3.2 ml. Canister 1 and canister 4 were stretched in the locked position while canister 2 and canister 3 remained unstretched.

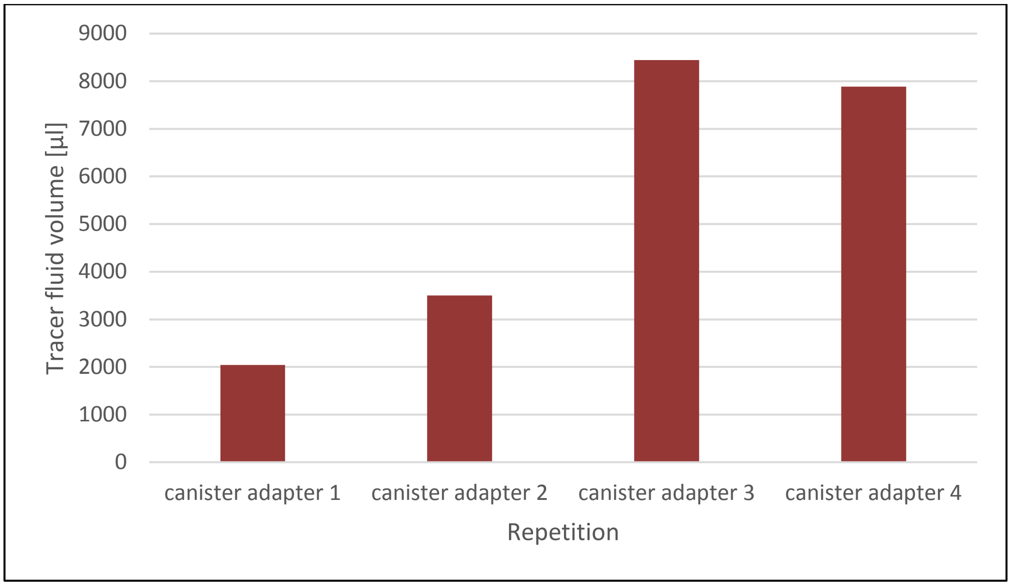

Figure 36: Measured residual quantity values at the canister adapter from four repetitions

The investigation has shown that, compared with the previously obtained exposure data, a relatively large amount of residue remains on the canister adapter even after rinsing. Since the canister adapter is openly accessible after unscrewing from the canister, an operator exposure at this point cannot be excluded. In the second part of the experiments, an exposure could also be detected in many cases when using the CTS. A high effectiveness in the rinsing is therefore a prerequisite for the operator protection. However, the measured values from the operator exposure trials are significantly lower than the residual quantities determined. In practice, therefore, the operator only is exposed to a very small proportion of the total residual quantity. It therefore must be noted that a rinsing test in this form cannot replace the actual exposure measurements.

4 Conclusions for testing of CTS

The trials have shown that most of the total exposure during the filling process accumulates on the protective gloves and overalls. In particular, the protective gloves are of great importance, because there is direct contact between the operator's hands and the components of the CTS so that an exposure of the protective gloves during the filling process is particularly probable. The primary focus of an examination should therefore be on the protective gloves. The exposure of visors, laboratory gloves and underwear is in proportion to that very low for all variants. In order to keep the costs and the time required for the test procedure as low as possible, it is therefore possible to dispense with the evaluation of these dosimeters.

For the assessment of CTS, objective criteria must be established in the form of permitted exposure limits, which do not have to be exceeded. Concerning the developed methods, it is crucial that these are precisely quantifiable in order to be able to determine without any doubt that the permitted exposure limits are met.

The exposure limit values therefore always have to be above the quantification limit of this type of dosimeter. For the overall, the following minimum condition applies:

$$G_{overall} > LOQ_{overall} \approx 9.78\ \mu l$$

$G_{overall}$ = Exposure limit value for testing of the overall
$LOQ_{overall}$ = Limit of quantification of the overall

Accordingly applies to the limit on the individual glove:

$$G_{glove} > LOQ_{glove} \approx 1.01 \mu l$$

G_{glove} = Exposure limit value for testing of the gloves
LOQ_{glove} = Limit of quantification of the glove

Since the result value for the plant protection gloves is the sum of the left and right glove, while the limit of quantification relates only to one glove, the special case that the measured value of one glove is below the limit of quantification has to be taken into account. The resulting higher uncertainty of the measurement result can be taken into account by an additional safety coefficient at the exposure limit for example.

In line with the assessment of drift reduction (Rautmann et al. 1999) (Herbst et al. 2012), standard reference values and associated reduction classes can also be defined for the assessment of CTS. The standard reference value can be derived from the trial variants without CTS. In the case of the protective gloves, the value of the 75th percentile at the induction hopper, the value of the 75th percentile at the dome shaft or a combination of both variants can be used. In order to be able to test without additional aids such as a stepladder, it may be useful to limit the tests in the height range of the induction hopper. With the resulting standard reference value of the induction hopper $S_{glove,I} = 14{,}16\ \mu l$, a reduction factor r can then be determined. The absolute value of the reduction factor is limited to an arithmetically achievable maximum value due to the above restriction of the limit.

$$r_{max,I} = 1 - \frac{G_{glove}}{S_{glove,I}} = 1 - \frac{1.01 \mu l}{14.16 \mu l} = 0.929$$

$r_{max,I}$ = Maximum reduction factor for induction hopper as standard reference
G_{glove} = Exposure limit value for testing of the gloves
$S_{glove,I}$ = Standard reference value of the induction hopper

The calculation of the maximum quantifiable reduction factor shows that, for example, an assessment for a 90 % reduction with the selected standard reference value is theoretically possible. It should be noted at this point, however, that the calculation presented here contains no safety coefficient. Since the blank values as well as the limits of detection and quantification of the measuring method can shift slightly, it is necessary to provide appropriate safety when defining the limit values, which reduces the reduction factor accordingly.

The determination of a reduction factor according to the above scheme is currently not useful for the overalls, since the standard reference values based on the 75th percentile are all below the limit of quantification and thus below the limit $G_{overall}$. The quantification limit would have to be reduced even more, in order to lower the quantifiable limit sufficiently far enough. This is hardly feasible under the current circumstances. Nevertheless, in order to be able to incorporate the overall's exposure into the evaluation, instead of the 75th percentile, the accumulated sum of the exposure measurement values from all repetitions could be used as an alternative, and a limit value could be formed from this. However, outliers would not be separated and measured values between the detection and quantification limits would be offset by an increased uncertainty. Measured values below the detection limit would not be charged. Nevertheless, such an additional criterion could be used in addition to the review of the Protective gloves to ensure that the exposure of the overall is also limited by the CTS to be tested.

5 Summary

The measuring method used to measure operator exposure during the filling process has been significantly improved by several innovative measures, such as a better washing program with less water, the operation of the washing machine with demineralized water, the use of stainless steel sieves for sample preparation and new overalls. In particular, during the test period, valuable experience was gained with the correct textile composition, the correct storage of the contaminated dosimeters and the evaluation of the measured data. The complex process could be made more efficient by new ideas in many places, so that a faster processing of the trials is possible. The measurement accuracy was improved or at least maintained depending on the dosimeter. Due to the independent filling of the tracer solution in PPP canisters, short storage times between filling and the beginning of the test could be realized, whereby the stability of the tracer and thus also the measuring accuracy are positively influenced. Especially in the variants without the CTS, the results show in some cases larger situation-dependent differences in the exposure of different operators. The execution of the tests should therefore be carried out by several operators with a sufficient number of repetitions. It can be deduced from the trial results that visors, laboratory gloves and underwear can largely be dispensed with when evaluating a CTS, since the proportion of these dosimeters in the total exposure is negligible. For overalls, the particular problem is that only a few valid measurements above the detection limit are available, although appreciable exposures may occur. On the other hand, the distribution of measurement results in protective gloves allows the derivation of a suitable standard reference value and is well suited as a basis for evaluating the filling systems. Due to the limits of quantification of the dosimeters, however, restrictions in the measuring methodology are set, which must not be disregarded when defining the test requirements.

6 References

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7 Appendix

Legend Measurement Tables

Legend measurement values

Style	Example value	Explanation (Assignment)
Italic	*0.00*	Value below LOD (Zero)
Bold	**1.11**	Value between LOD and LOQ (Mean of LOD and LOQ)
Regular	2.22	Value higher than LOQ
Asterisk	3.33*	Mismeasurement (no evaluation)

Measurements Part 1

Protective Visor

Operator	Tracer fluid volume (µl)		
	1 liter	5 liter	10 liter
O1	*0.00*	*0.00*	*0.00*
O2	*0.00*	*0.00*	*0.00*
O3	*0.00*	0.20	*0.00*
O4	*0.00*	*0.00*	*0.00*
O5	*0.00*	*0.00*	*0.00*
O6	*0.00*	*0.00*	*0.00*
O7	*0.00*	*0.00*	*0.00*
Average	0.00	0.03	0.00
Standard deviation	0.00	0.08	0.00
75th-percentile	0.00	0.00	0.00

Protective gloves

Operator	Tracer fluid volume (µl)		
	1 liter	5 liter	10 liter
O1	4.29	8.02	32.34
O2	11.65	16.60	11.51
O3	9.07	11.51	8.46
O4	0.68	2.83	51.65
O5	4.09	7.57	1.62
O6	5.20	6.11	2.47
O7	24.61	12.58	4.52
Average	8.51	9.32	16.08
Standard deviation	7.95	4.58	18.87
75th-percentile	10.36	12.05	21.93

Laboratory gloves

Operator	Tracer fluid volume (µl)		
	1 liter	5 liter	10 liter
O1	*0.00*	*0.00*	*0.00*
O2	*0.00*	*0.00*	*0.00*
O3	*0.00*	*0.00*	*0.00*
O4	*0.00*	*0.00*	*0.00*
O5	*0.00*	*0.00*	*0.00*
O6	*0.00*	*0.00*	*0.00*
O7	*0.00*	*0.00*	*0.00*
Average	0.00	0.00	0.00
Standard deviation	0.00	0.00	0.00
75th-percentile	0.00	0.00	0.00

Protective overalls

Operator	**Tracer fluid volume (µl)**		
	1 liter	**5 liter**	**10 liter**
O1	*0.00*	11.48	17.59
O2	*0.00*	3.96	*0.00*
O3	*0.00*	*0.00*	5.26
O4	*0.00*	*0.00*	*0.00*
O5	*0.00*	*0.00*	*0.00*
O6	*0.00*	*0.00*	*0.00*
O7	*0.00*	*0.00*	*0.00*
Average	0.00	2.21	3.26
Standard deviation	0.00	4.35	6.62
75th-percentile	0.00	1.98	2.63

Long underwear

Operator	**Tracer fluid volume (µl)**		
	1 liter	**5 liter**	**10 liter**
O1	*0.00*	*0.00*	1.37
O2	0.98	*0.00*	*0.00*
O3	*0.00*	0.93	*0.00*
O4	*0.00*	2.75	*0.00*
O5	1.05	0.85	1.12
O6	*0.00*	0.78	*0.00*
O7	1.30	*0.00*	*0.00*
Average	0.47	0.76	0.36
Standard deviation	0.60	0.98	0.61
75th-percentile	1.01	0.89	0.56

Field Recovery - Overall + Underwear

Operator	Recovery rate (%)
O1	72.91
O2	94.86
O3	77.55
O4	91.02
O5	93.53
O6	77.82
O7	88.65
Average	85.19
Standard deviation	8.87

Field Recovery - Protective gloves

Operator	Recovery rate (%)
O1	103.50
O2	102.42
O3	105.74
O5	87.74
Average	99.85
Standard deviation	8.19

Measurements Part 2

Protective Visor

Operator	Repetition no.	Tracer fluid volume (µl)			
		Induction hopper	**Induction hopper with CTS**	**Dome shaft**	**Dome shaft with CTS**
O1	1	*0.00*	*0.00*	*0.00*	*0.00*
	2	*0.00*	*0.00*	*0.00*	*0.00*
	3	*0.00*	*0.00*	*0.00*	*0.00*
	4	*0.00*	*0.00*	*0.00*	*0.00*
	5	*0.00*	*0.00*	*0.00*	*0.00*
	Average	0.00	0.00	0.00	0.00
	Standard dev.	0.00	0.00	0.00	0.00
O2	1	*0.00*	*0.00*	*0.00*	*0.00*
	2	*0.00*	*0.00*	*0.00*	*0.00*
	3	*0.00*	*0.00*	*0.00*	*0.00*
	4	*0.00*	*0.00*	*0.00*	*0.00*
	5	*0.00*	*0.00*	*0.00*	*0.00*
	Average	0.00	0.00	0.00	0.00
	Standard dev.	0.00	0.00	0.00	0.00
O8	1	*0.00*	*0.00*	*0.00*	*0.00*
	2	*0.00*	*0.00*	*0.00*	*0.00*
	3	*0.00*	*0.00*	*0.00*	*0.00*
	4	*0.00*	*0.00*	*0.00*	0.20
	5	*0.00*	*0.00*	*0.00*	0.20
	Average	0.00	0.00	0.00	0.08
	Standard dev.	0.00	0.00	0.00	0.11
Average		0.00	0.00	0.00	0.03
Standard deviation		0.00	0.00	0.00	0.07
75th-percentile		0.00	0.00	0.00	0.00

Protective gloves

Operator	Repetition no.	Tracer fluid volume (µl)			
		Induction hopper	**Induction hopper with CTS**	**Dome shaft**	**Dome shaft with CTS**
O1	1	11.11	0.66	11.45	*0.00*
	2	14.31	0.66	12.91	2.31
	3	8.01	2.55	13.40	*0.00*
	4	10.50	1.89	8.12	*0.00*
	5	31.16	0.66	15.75	0.68
	Average	15.02	1.28	12.33	0.60
	Standard dev.	9.30	0.88	2.81	1.00
O2	1	3.76	*0.00*	13.91	0.65
	2	16.50	*0.00*	14.71	1.85
	3	6.87	*0.00*	8.83	0.65
	4	21.90	*0.00*	12.26	0.65
	5	14.01	0.55	34.90	*0.00*
	Average	12.61	0.11	16.92	0.76
	Standard dev.	7.33	0.25	10.30	0.67
O8	1	7.26	*0.00*	42.50	*0.00*
	2	5.03	*0.00*	76.59	*0.00*
	3	7.90	*0.00*	20.38	1.37
	4	3.87	0.69	33.72	4.24*
	5	7.50	0.69	23.73	0.68
	Average	6.31	0.28	39.38	0.51
	Standard dev.	1.76	0.38	22.54	0.65
Average		11.31	0.56	22.88	0.63
Standard deviation		7.44	0.76	18.10	0.75
75th-percentile		14.16	0.68	28.72	0.68

Laboratory gloves

Operator	Repetition no.	Tracer fluid volume (µl)			
		Induction hopper	Induction hopper with CTS	Dome shaft	Dome shaft with CTS
O1	1	*0.00*	*0.00*	*0.00*	*0.00*
	2	*0.00*	*0.00*	*0.00*	*0.00*
	3	*0.00*	*0.00*	*0.00*	*0.00*
	4	0.32	*0.00*	*0.00*	*0.00*
	5	*0.00*	*0.00*	*0.00*	*0.00*
	Average	0.06	0.00	0.00	0.00
	Standard dev.	0.14	0.00	0.00	0.00
O2	1	*0.00*	*0.00*	*0.00*	*0.00*
	2	*0.00*	*0.00*	*0.00*	*0.00*
	3	*0.00*	*0.00*	*0.00*	*0.00*
	4	*0.00*	*0.00*	*0.00*	*0.00*
	5	*0.00*	*0.00*	*0.00*	*0.00*
	Average	0.00	0.00	0.00	0.00
	Standard dev.	0.00	0.00	0.00	0.00
O8	1	*0.00*	*0.00*	0.16	*0.00*
	2	*0.00*	*0.00*	*0.00*	*0.00*
	3	*0.00*	*0.00*	*0.00*	*0.00*
	4	*0.00*	*0.00*	*0.00*	*0.00*
	5	*0.00*	*0.00*	*0.00*	*0.00*
	Average	0.00	0.00	0.03	0.00
	Standard dev.	0.00	0.00	0.07	0.00
Average		0.02	0.00	0.01	0.00
Standard deviation		0.08	0.00	0.04	0.00
75th-percentile		0.00	0.00	0.00	0.00

Protective overalls

Operator	Repetition no.	Tracer fluid volume (µl)			
		Induction hopper	**Induction hopper with CTS**	**Dome shaft**	**Dome shaft with CTS**
O1	1	23.36	*0.00*	*0.00*	15.29
	2	60.80	*0.00*	*0.00*	*0.00*
	3	62.49	*0.00*	*0.00*	*0.00*
	4	12.36	9.90	*0.00*	*0.00*
	5	6.07	4.92	*0.00*	*0.00*
	Average	33.02	2.96	0.00	3.06
	Standard dev.	26.86	4.42	0.00	6.84
O2	1	*0.00*	*0.00*	*0.00*	9.02
	2	*0.00*	*0.00*	*0.00*	*0.00*
	3	*0.00*	*0.00*	*0.00*	16.41
	4	4.59	*0.00*	*0.00*	*0.00*
	5	*0.00*	*0.00*	*0.00*	5.44
	Average	0.92	0.00	0.00	6.17
	Standard dev.	2.05	0.00	0.00	6.89
O8	1	*0.00*	*0.00*	*0.00*	*0.00*
	2	*0.00*	*0.00*	8.85	*0.00*
	3	*0.00*	*0.00*	9.88	*0.00*
	4	*0.00*	*0.00*	32.31	*0.00*
	5	*0.00*	*0.00*	*0.00*	*0.00*
	Average	0.00	0.00	10.21	0.00
	Standard dev.	0.00	0.00	13.22	0.00
Average		11.31	0.99	3.40	3.08
Standard deviation		21.45	2.77	8.64	5.81
75th-percentile		9.21	0.00	0.00	2.72

Long underwear

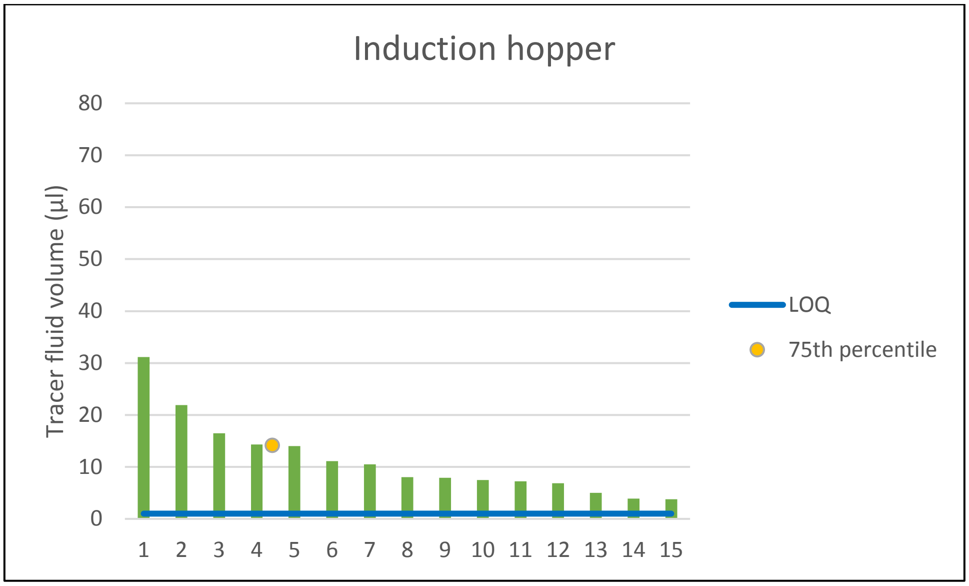
Operator	**Repetition no.**	**Tracer fluid volume (µl)**			
		Induction hopper	**Induction hopper with CTS**	**Dome shaft**	**Dome shaft with CTS**
O1	1	*0.00*	3.97	*0.00*	*0.00*
	2	1.03	*0.00*	*0.00*	*0.00*
	3	*0.00*	4.79	*0.00*	*0.00*
	4	*0.00*	3.91	*0.00*	1.21
	5	2.78	5.10	81.51*	*0.00*
	Average	0.76	3.56	0.00	0.24
	Standard dev.	1.21	2.05	0.00	0.54
O2	1	3.57	*0.00*	*0.00*	*0.00*
	2	*0.00*	*0.00*	*0.00*	*0.00*
	3	*0.00*	*0.00*	*0.00*	*0.00*
	4	*0.00*	*0.00*	*0.00*	3.85
	5	*0.00*	*0.00*	*0.00*	*0.00*
	Average	0.71	0.00	0.00	0.77
	Standard dev.	1.60	0.00	0.00	1.72
O8	1	*0.00*	*0.00*	*0.00*	*0.00*
	2	*0.00*	*0.00*	*0.00*	*0.00*
	3	*0.00*	*0.00*	*0.00*	*0.00*
	4	*0.00*	*0.00*	*0.00*	*0.00*
	5	*0.00*	*0.00*	*0.00*	*0.00*
	Average	0.00	0.00	0.00	0.00
	Standard dev.	0.00	0.00	0.00	0.00
Average		0.49	1.19	0.00	0.34
Standard deviation		1.13	2.05	0.00	1.02
75th-percentile		0.00	1.96	0.00	0.00

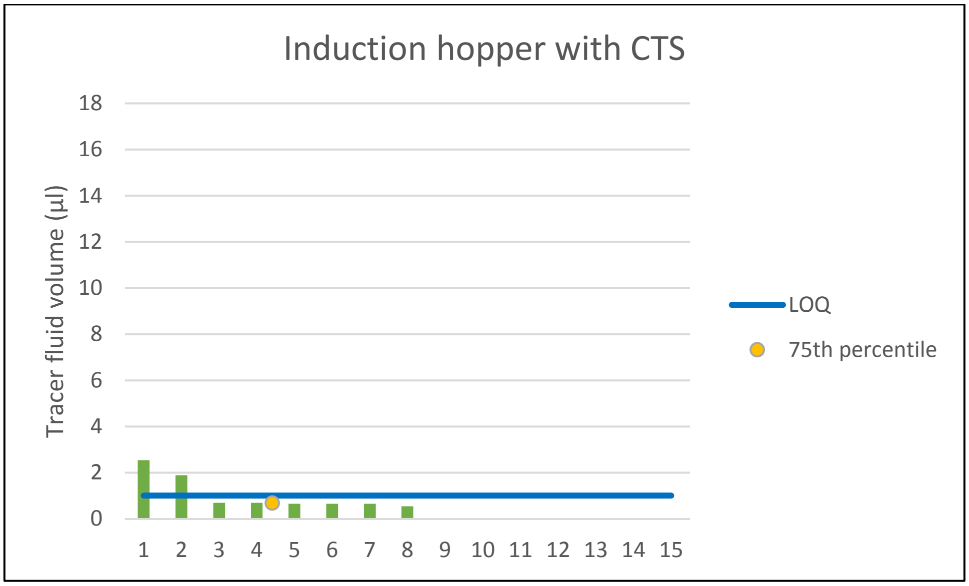
Field Recovery - Overall + Underwear

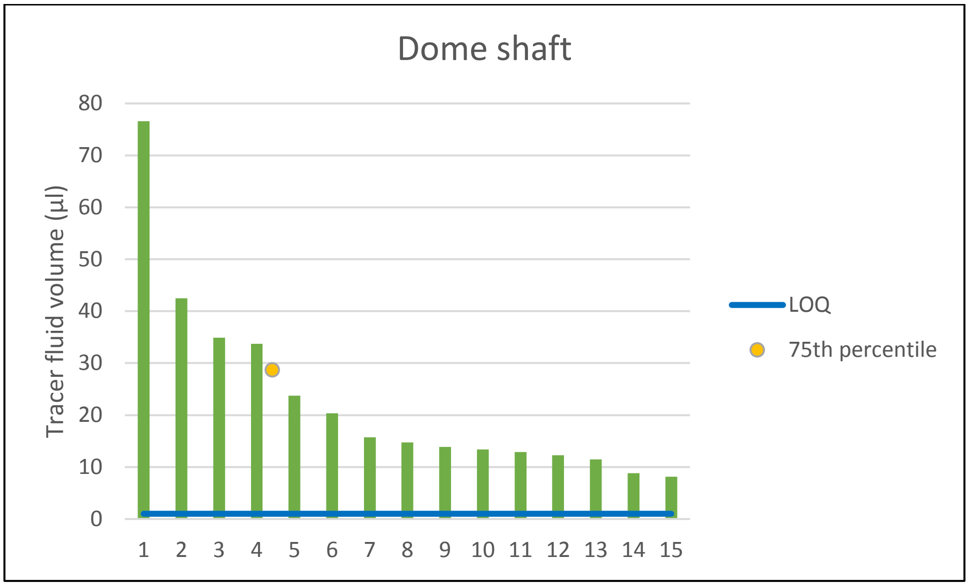
Operator-Setup	Recovery rate (%)
O1-I	92.79
O1-ICTS	55.87
O1-D	131.83
O1-DCTS	74.69
O2-I	98.74
O2-ICTS	73.33
O2-D	48.09
O2-DCTS	46.73
O8-I	98.90
O8-ICTS	69.41
O8-D	92.05
O8-DCTS	102.32
Average	82.06
Standard deviation	25.30

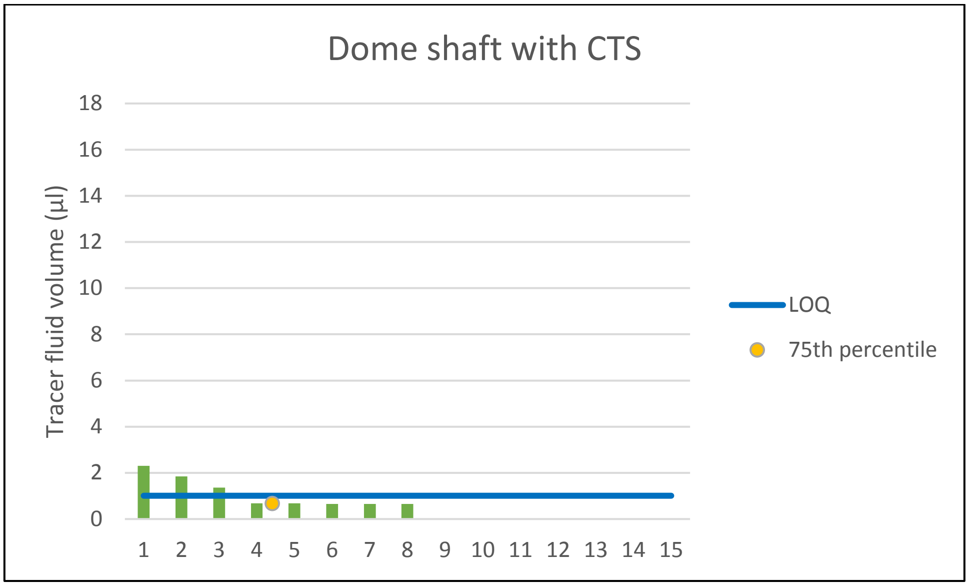
Diagrams

Distribution of measurements – Protective gloves

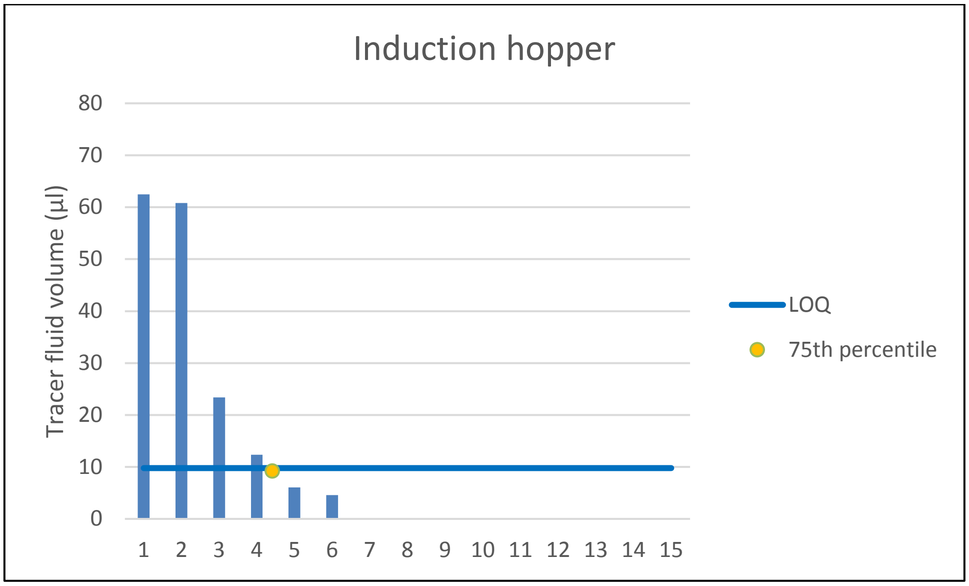


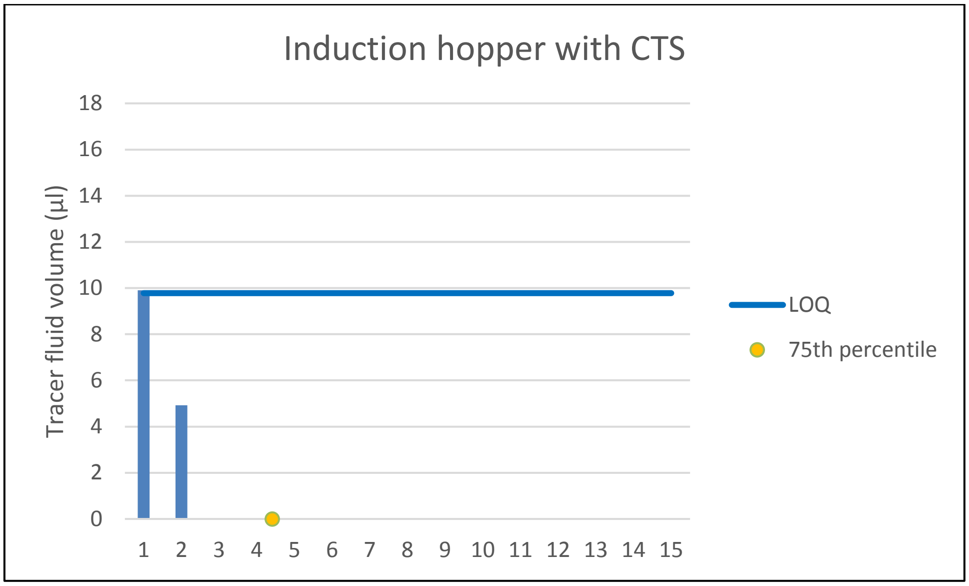


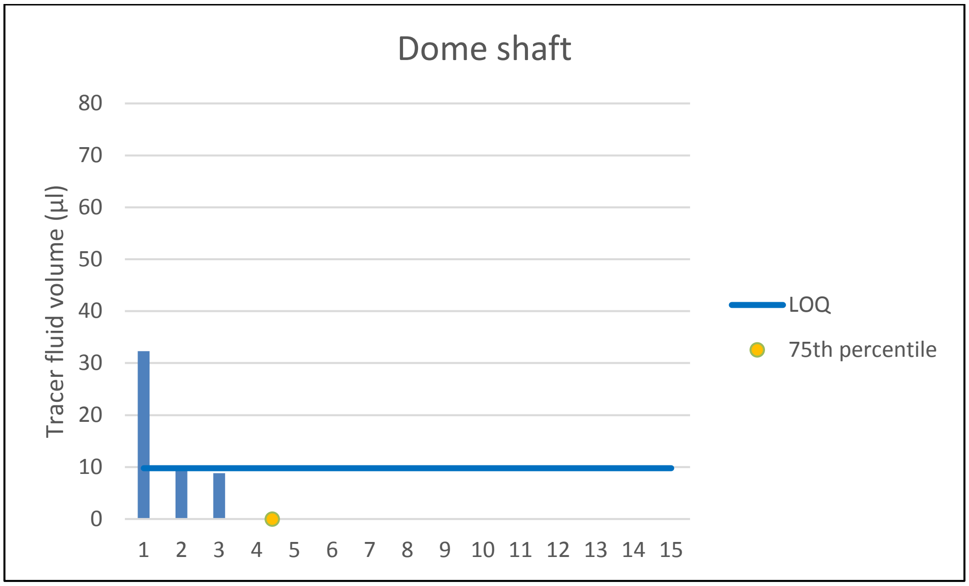

Dome shaft
Tracer fluid volume (µl)
80
70
60
50
40
30
20
10
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
LOQ
75th percentile

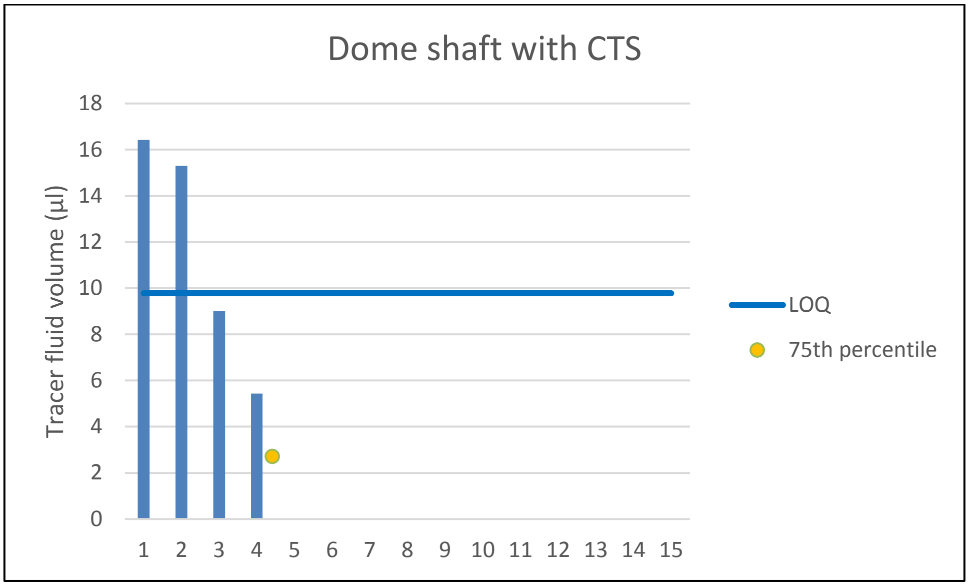

Dome shaft with CTS
Tracer fluid volume (µl)
18
16
14
12
10
8
6
4
2
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
LOQ
75th percentile

Distribution of measurements – Protective overalls






Dome shaft
Tracer fluid volume (µl)
80
70
60
50
40
30
20
10
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
LOQ
75th percentile


Dome shaft with CTS
Tracer fluid volume (µl)
18
16
14
12
10
8
6
4
2
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
LOQ
75th percentile

„Berichte aus der Biologischen Bundesanstalt für Land- und Forstwirtschaft“
erscheinen seit 1995 in zwangloser Folge

Seit 2008 werden sie unter neuem Namen weitergeführt:
„Berichte aus dem Julius Kühn-Institut“

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Heft 177, 2014	Siebentes Nachwuchswissenschaftlerforum 2014, 26. - 28. November in Quedlinburg - Abstracts -, 57 S.
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Julius Kühn-Institut
Bundesforschungsinstitut für Kulturpflanzen