

US EPA ARCHIVE DOCUMENT

SANDOZ AGRO LTD.
DEVELOPMENT REGION BASLE
ANALYTICAL CHEMISTRY AND ENVIRONMENTAL SCIENCES

TDS-No.: BS 1988

TABLE OF CONTENTS

	SUMMARY	Page
		3
1.	INTRODUCTION	4
2.	REAGENTS	4
2.1.	STANDARD SOLUTIONS	
2.1.1.	STOCK SOLUTIONS OF STANDARDS	4
2.1.2.	WORKING SOLUTIONS OF STANDARDS	4
3.	APPARATUS	5
4.	PROCEDURE	
4.1.	SAMPLE PREPARATION	6
4.1.1.	CORN GRAIN	6
4.1.2.	STRAW	6
4.1.3.	CORN PLANT AND COB	6
4.1.4.	SOIL	6
4.2.	EXTRACTION	
4.2.1.	CORN GRAIN OR COB	6
4.2.2.	CORN STRAW AND CORN PLANT (DRY)	6
4.2.3.	CORN PLANT (GREEN)	7
4.2.4.	SOIL	7
4.3.	CLEAN UP I	
4.3.1.	CONDITIONING OF THE PRE-PACKED MEGA-BOND ELUT COLUMN	7
4.3.2.	CLEAN UP PROCEDURE	7
4.4.	CLEAN UP II	
4.4.1.	SILICA GEL COLUMN PREPARATION	7
4.4.2.	CLEAN UP PROCEDURE	7
4.5.	GAS CHROMATOGRAPHIC ANALYSIS	8
4.5.1.	CHECKING THE DETECTOR RESPONSE	
4.5.1.1.	TEST OF VARIANCE HOMOGENEITY	8
4.5.1.2.	TEST THE LINEARITY OF THE DETECTOR RESPONSE	8
4.5.2.	GAS CHROMATOGRAPHIC CONDITIONS	9
5.	QUANTIFICATION	
5.1.	ANALYSIS SEQUENCE/CALIBRATION FUNCTION	11
5.2.	DETERMINATION OF THE ANALYSIS CONCENTRATION IN A SAMPLE	11
5.4.	LIMIT OF DETERMINATION/DETECTION	11
6.	RESULTS AND DISCUSSION	
	VALIDATION DATA	13

SANDOZ AGRO LTD.
DEVELOPMENT REGION BASLE
ANALYTICAL CHEMISTRY AND ENVIRONMENTAL SCIENCES

TDS-No.: BS 1988

A METHOD FOR THE DETERMINATION OF THE RESIDUES OF SAN 582 H IN CORN AND SOIL SAMPLES

SUMMARY

The method has been developed for the determination of SAN 582 H in corn (whole plant, grain, cobs and straw) and in soil samples and was validated in the concentration range from 0.01 mg/kg to 0.6 mg/kg.

SAN 582 H is extracted with two 250 ml portions of methanol:water (95:5). The combined organic extracts are cleaned up by solid phase extraction on a pre-packed reversed phase C-18 column. Further clean up is achieved by partition between water and toluene followed by chromatography on a silica gel column using increasing volumes of ethylacetate:cyclohexane (2:8) for pre-wash and elution. The eluate is concentrated and dissolved in toluene. Separation and quantification is performed by capillary column gas chromatography using a thermoionic nitrogen specific detector (TSD).

The limit of detection is <0.01 mg/kg for all crop matrices and < 0.005 mg/kg for soil matrices analyzed. The limit of determination depends on weight of the sample aliquot used for analysis and on the actual linearity of the detector response. It was found to be 0.01 mg/kg or lower for crop matrices and 0.005 mg/kg or lower for soil matrices (assuming a 95% confidence interval). Therefore the limit of determination was defined to be 0.01 mg/kg for crop and 0.005 mg/kg for soil matrices.

The average recovery was $101 \pm 13\%$ (n=8, fortification range 0.01-0.6 mg/kg) for whole plant, $97 \pm 8\%$ (n=7, fortification range 0.02 - 0.06 mg/kg) for straw, $97 \pm 2\%$ (n=5, fortification range 0.02-0.3 mg/kg) for grain and cob and $102 \pm 8\%$ (n=6, fortification range 0.01 - 0.27 mg/kg) for soil.

SANDOZ AGRO LTD.
DEVELOPMENT REGION BASLE
ANALYTICAL CHEMISTRY AND ENVIRONMENTAL SCIENCES

TDS-No.: BS 1988

1. INTRODUCTION

The objective of residue methods is to determine the concentration of weathered residues in crop and soil samples with the highest possible sensitivity. Therefore this method uses Gas Liquid Chromatography in combination with a Nitrogen specific detector. These conditions were selected in order to achieve a limit of determination as low as possible.

2. REAGENTS

Methanol, cyclohexane, ethyl acetate, toluene and hexadecane -- pesticide grade or redistilled in glass.

Celite 545

Potassium permanganate (Code No. 5082; E. Merck) Darmstadt, Federal Republic of Germany)

EXTRELUT[®] column -- pre-packed or prepared with EXTRELUT[®] Kieselguhr (Code No. 11737 and 11738; E. Merck¹)

MEGA-BOND-ELUT C-18 pre-packed solid phase extraction columns (10 g, 60 ml, Analytichem International, USA, Code-No.: A 1225-6031)

Silica gel 60, 230 - 400 mesh ASTM (Code No. 9385; E. Merck)

Sea sand, acid-washed (Code No. 7712; E. Merck)

2.1. STANDARD SOLUTIONS

2.1.1. STOCK SOLUTIONS OF STANDARDS

Dissolve 100 mg of SAN 582 H analytical standard of known purity in methanol and make up to 10 ml with toluene. This gives a 10 mg/ml standard solution (10000 µg/ml). Store the solution tightly stoppered at - 20 °C. Prepare fresh solution every 12 months.

2.1.2. WORKING SOLUTIONS OF STANDARDS

Transfer 1 ml of the stock solution into a volumetric flask and make up to 10 ml with toluene. This gives a 1 mg/ml (1000 µg/ml) standard solution. Transfer 1 ml of the 1000 µg/ml stock solution to a 100 ml volumetric flask and make up to volume with toluene to get a standard solution containing 10 µg/ml. Prepare a range of standards for the GC/TSD quantification by diluting aliquots from the latter solutions with toluene (concentration range: 0.01 µg/ml to 1 µg/ml). Dilutions larger than 1:100 may not be used. Store the standard solutions at temperatures below 5 °C.

The working solutions will also be used for fortifying check samples.

1. Published by E. Merck, Darmstadt: "EXTRELUT, Neues Verfahren zur Extraktion lipophiler Stoffe", 1977.

SANDOZ AGRO LTD.
DEVELOPMENT REGION BASLE
ANALYTICAL CHEMISTRY AND ENVIRONMENTAL SCIENCES

TDS-No.: BS 1988

3. APPARATUS

Ultracentrifugal grinder	Model Z M 1, K. Reutsch AG, Haan (FRG)
Chopper	Kitchencutter, Hobart Swiss AG
Cutter	Nakocutter, Hobart Swiss AG
Blender	Turmix, Küssnacht, Switzerland
Platform shaker	Infors AG, Basel, Switzerland
Rotary vacuum evaporator	Büchi Flavil, Switzerland
Mettler balance*	PL 3000
Mettler balance*	AK 160
Screw-cap bottles	1 L capacity
Filtering flasks	
Büchner funnels	
Dropping funnels	50 ml, graduated
Measuring cylinders	100 ml
Filter paper	Macherey-Nagel & Co.; No. MN 640 m
Erlenmeyer flasks	100 ml
Chromatographic tubes for clean up	200 mm x 25 mm (i.d.) glass column with ground glass top
Separatory funnels	250 ml
Round bottom flasks	1 L, 500 ml, 100 ml
Pipettes	1 - 10 ml
Gas chromatograph*	Vista 6000, with a thermoionic nitrogen specific detector (TSD) or Varian GC 3400, with a thermoionic nitrogen specific detector (TSD)
Automatic sampler*	Varian Autosampler 8000 Varian Autosampler 8035
Sampling vials	10 µl, Varian
Microliter syringes	1 µl, Linear 585, Burkard Instrumente AG, Zürich, Switzerland
Recorder	
Laboratory data processing system*	Varian, Vista 402
Gas chromatographic columns	see 4.5.

* Other models equivalent to those mentioned here may be used.

SANDOZ AGRO LTD.
DEVELOPMENT REGION BASLE
ANALYTICAL CHEMISTRY AND ENVIRONMENTAL SCIENCES

TDS-No.: BS 1988

4. PROCEDURE

4.1. SAMPLE PREPARATION

General: The size of laboratory samples depends on the matrix sampled and on the growth stage at date of sampling. Analytical samples for extraction are prepared from the entire laboratory sample. This is achieved by cutting - if applicable - and mixing the complete material of the laboratory sample prior to sub-sampling for extraction.

4.1.1. CORN GRAIN

Grind about 200 g grain in the ultracentrifugal grinder. Weigh out 100 g of the grain powder for extraction.

4.1.2. STRAW

Cut the straw into 10-20 mm pieces or chop it in a food chopper. Mix well and weigh out 25 g for extraction.

4.1.3. CORN PLANT AND COB

Cut the plant material into 10-20 mm pieces or chop it in a food chopper. Mix well and weigh out 100 g for extraction.

4.1.4. SOIL

Mix the soil sample thoroughly, remove stones larger than 1 cm in diameter. Determine the moisture content of the soil as follows: weigh an aliquot (100-200 g), air-dry the sample overnight at room temperature and weigh again. Determine the factor

$$f_w = \frac{\text{g soil un-dried}}{\text{g soil air-dried}}$$

to be included in the final calculation of the soil dry weight used for the extraction.

4.2. EXTRACTION

4.2.1. CORN GRAIN OR COB

Transfer 100 g of the grain powder or cut cobs into a blender jar, add 20 g Celite and ca. 250 ml methanol:water (95:5) and blend at high speed for ca. three minutes. Filter under suction.

Transfer the filter cake into a 1 L screw cap bottle and shake with ca. 250 ml methanol:water (95:5) for ca. 30 min. Filter as before and discard the filter cake. Combine the filtrates and make up to a definite volume with methanol:water (95:5). Take 1/4 of the latter extract (= 25 g plant material) and add twice its volume of water so as to obtain a methanol:water ratio of approximately 2:1. 1:2

4.2.2. CORN STRAW AND CORN PLANT (DRY)

Transfer 25 - 50 g (dependent on the state of dryness) of the material into a blender jar, add 20 g Celite and ca. 300 ml methanol:water (95:5) and blend at high speed for ca. three minutes. Filter under suction.

Transfer the filter cake into a 1 L screw cap bottle and shake with ca. 250 ml methanol:water (95:5) for ca. 30 min. Filter as before, discard the filter cake and combine the filtrates. Evaporate the combined filtrates to ca. 300 ml and make up to a definite volume with methanol:water (95:5). Take an aliquot from the extract solution corresponding to 25 g of plant material and add twice its volume of water so as to obtain a methanol:water ratio of approximately 2:1. 1:2

SANDOZ AGRO LTD.
DEVELOPMENT REGION BASLE
ANALYTICAL CHEMISTRY AND ENVIRONMENTAL SCIENCES

TDS-No.: BS 1988

4.2.3. CORN PLANT (GREEN)

Transfer 100 g of the material into a blender jar, add 20 g Celite and ca. 250 ml methanol:water (95:5) and blend at high speed for ca. three minutes. Filter under suction.

Transfer the filter cake into a 1 L screw cap bottle and shake with ca. 200 ml methanol for 30 minutes. Filter under suction and discard the filter cake. Combine the filtrates and make up to a definite volume with methanol:water (95:5). Take 1/4 of the latter extract (= 25 g plant material) and add twice its volume of water so as to obtain a methanol:water ratio of approximately 2:1. 1:2

4.2.4. SOIL

Transfer at least 100 g of the un-dried soil sample into a blender jar, add 20 g Celite and ca. 250 ml methanol:water (95:5) and blend at high speed for ca. three minutes. Filter under suction.

Transfer the filter cake into a 1 L screw cap bottle and shake with ca. 250 ml methanol:water (95:5) for ca. 30 min. Filter as before and discard the filter cake. Combine the filtrates and make up to a definite volume with methanol:water (95:5). Take 1/2 of the latter extract (= 50 g soil) and add twice its volume of water so as to obtain a methanol:water ratio of approximately 2:1. 1:2

4.3. CLEAN UP I

4.3.1. CONDITIONING OF THE PRE-PACKED MEGA-BOND ELUT COLUMN

Wash the Mega-Bond Elut column by suction successively with 1 volume (= 60 ml) of methanol and 1 volume (= 60 ml) of methanol/water 1:1. Do not let the column dry.

4.3.2. CLEAN UP PROCEDURE

Pass the extract from 4.2. through the prepared column by applying vacuum (150-250 mbar) until the solvent reaches the top layer of the solid phase material. Wash the column with 2 volumes (120 ml) methanol/water 1:1. Discard the washings. Subsequently elute the column with 1 volume (60 ml) methanol/water 85:15.

Evaporate the eluate at 40° C under reduced pressure (150-250 mbar) until the methanol is removed and only water remains.

4.4. CLEAN UP II

4.4.1. SILICA GEL COLUMN PREPARATION

Place a plug of cotton at the bottom of a chromatographic tube, which can withstand a pressure of about two atm. Add to the column successively sea sand (1 cm layer), silica gel (15 g) and again sea sand (1 cm layer). Add 80 ml of toluene to the column and press (using compressed air at a pressure of about 0.1 atm) the solvent down through the column until its level is flush with the top of the upper layer of sea sand. Repeat the process if necessary, until the silica gel is translucent.

4.4.2. CLEAN UP PROCEDURE

Transfer the aqueous remainder from the evaporation of step 4.3.2. into a 50 ml graduated dropping funnel mounted over an Extrelut² column which in turn is mounted over the silica gel column (Figure 1). Wash the flask that contained the residues with small amounts of water and transfer the washings into the dropping funnel until the total volume is 20 ml. Let the solution flow into the Extrelut² column and wait for about 10 minutes so that the solution is well adsorbed on the column. Add 100 ml of toluene to the flask used for evaporation, swirl well and transfer the washing through the dropping funnel into the Extrelut² column.

SANDOZ AGRO LTD.
DEVELOPMENT REGION BASLE
ANALYTICAL CHEMISTRY AND ENVIRONMENTAL SCIENCES

TDS-No.: BS 1988

PRE-WASH PROCEDURE:

Allow the eluate from the Extrelut[®] column to pass directly onto the silica gel column with the stop-cock of the silica gel column open and collect the eluate in a 500 ml round bottom flask. When the level of liquid reaches the top of the upper sea sand layer in the silica gel column, close the stop-cock and remove the EXTRELUT[®] column. Add 30 ml of a mixture of acetate/cyclohexane 2:8 into the silica gel column. Open the stop-cock press (using compressed air at a pressure of about 0.1 atm) the ethyl acetate/cyclohexane mixture through the silica gel column until the solvent level reaches the top of the upper layer of the sea sand. Remove the round bottom flask. Discard the collected eluates.

ELUTION:

Mount a 250 ml round bottom below the outlet of the column. Add 90 ml of ethyl acetate/cyclohexane 2:8 to the column and press (using compressed air at a pressure of about 0.1 atm) it through the column. Continue pressing until no more is collected. Add three drops of hexadecane to the collected solution. Evaporate the eluate just to dryness (rotary evaporator, 40°C, 10-20 mbar). Dissolve the residue in 3 ml of toluene, use the resulting solution directly for GC analysis.

4.5. GAS CHROMATOGRAPHIC ANALYSIS

Prior to each analysis sequence the GC response is checked for variance homogeneity and linearity.

4.5.1. CHECKING THE DETECTOR RESPONSE**4.5.1.1. TEST OF VARIANCE HOMOGENEITY**

Inject ten times standard solutions of the highest and the lowest working range level. Calculate the mean values and the corresponding standard deviations for both series. Calculate the test quantity TQ by dividing the squares of the two standard deviations (the variances):

$$TQ = s_{max}^2 / s_{min}^2 \quad (s_{max}^2 > s_{min}^2)$$

Compare TQ with the corresponding F-value (P=99%, $f_1 = n_{max} - 1$, $f_2 = n_{min} - 1$). If $TQ > F(99\%, f_1, f_2)$ then reduce the working range until the variances are homogeneous.

4.5.1.2. TEST THE LINEARITY OF THE DETECTOR RESPONSE

Verify that the detector response is linear over the working range and the retention time is stable. This is done by injecting standard solutions (preferably at least five different equidistant concentrations) prior to the analysis of the samples and after every two samples during analysis.

SANDOZ AGRO LTD.
DEVELOPMENT REGION BASLE
ANALYTICAL CHEMISTRY AND ENVIRONMENTAL SCIENCES

TDS-No.: BS 1988

5. QUANTIFICATION

5.1. ANALYSIS SEQUENCE/CALIBRATION FUNCTION

Load the automatic sampler with the cleaned-up extracts of the analytical samples and controls interspersed with the standards at selected concentrations. The standard solutions should consist of a set of at least five equidistant concentrations (covering the working range). Inject these solutions in duplicate into the equilibrated gas chromatograph by means of the automatic sampler. Measure peak heights/areas and determine the calibration function F from the corresponding peak heights/areas by means of a least squares regression procedure (Printouts and plots of typical calibration functions are given in tables 1 and 2, figure 2).

Determine the accuracy of F by dividing the mean residual sum of squares ' s_y ' by the mean value of the concentrations ' x ' to obtain ' v ':

$$v = s_y / x$$

where v is the variation coefficient. If v is larger than 0.1 then repeat the analysis series by using a larger number of standard levels.

5.2. DETERMINATION OF THE ANALYTES CONCENTRATION IN A SAMPLE

Calculate concentration of the residue in the extract by

$$\text{residue in extract (}\mu\text{g/ml)} = F^{-1} (\text{detector response extract})$$

where F is the multipoint calibration function and F^{-1} the inverse function.

Calculate the (corrected) residue level in a sample using the following formula (1):

$$\text{residue (mg/kg)} = \frac{\text{residue in extract (}\mu\text{g/ml)} \times \text{final volume (ml)} \times (100\%)}{\text{sample weight (g)} \times (\text{recovery } (\%))}$$

where the final volume includes dilution steps - if any.

5.3. DETERMINATION OF RECOVERIES

Calculate recoveries from fortified samples by comparing the chromatographic results from an untreated and a fortified sample. The following formula is used:

$$\text{Recovery} = \frac{A - C}{S} \times 100\%$$

where

A = concentration found in the fortified control sample (in $\mu\text{g/g}$)

C = concentration found in the control sample (in $\mu\text{g/g}$)

S = concentration added to the fortified control sample (in $\mu\text{g/g}$)

5.4. LIMIT OF DETERMINATION/DETECTION

The actual limits of determination and detection in units of the standard solutions are calculated from the calibration curve. The 95% confidence interval of the calibration function is determined using the pertinent statistical parameters (see tables 1 and 2). The limit of detection (L_1) is calculated as the lowest concentration being significantly different (50% confidence level) from Zero. The limit of determination (L_2) is calculated as the lowest concentration being significantly different (95% confidence level)

SANDOZ AGRO LTD.
DEVELOPMENT REGION BASLE
ANALYTICAL CHEMISTRY AND ENVIRONMENTAL SCIENCES

TDS-No.: BS 1988

from Zero. Figure 2 illustrates the procedure. The latter calculation is performed for each analysis sequence. The limits of detection or determination in mg/kg units are calculated from L_1 and L_2 , respectively, the sample weight used for analysis and the endvolume of the extract. The limits of detection were below 0.01 mg/kg or 0.005 mg/kg for crop and soil matrices, respectively in all instances, the actual value depends on the accuracy of the calibration curve. The corresponding limits of determination ranged from 0.001 to 0.01 mg/kg and from 0.0005 to 0.005 mg/kg for crop and soil matrices, respectively.

The limit of determination of the method therefore can be set to 0.01 mg/kg for crop and to 0.005 mg/kg for soil matrices.

SANDOZ AGRO LTD.
DEVELOPMENT REGION BASLE
ANALYTICAL CHEMISTRY AND ENVIRONMENTAL SCIENCES

TDS-No.: BS 1988

6. RESULTS AND DISCUSSION

VALIDATION DATA

The method was validated by performing a series of 26 fortification experiments in four different sample matrices.

These experiments were carried out by fortifying untreated samples with known amounts of SAN 582 H prior to extraction. Both, the untreated as well as the fortified samples were carried through the analytical procedure described above. Spiking levels ranged from 0.01-0.6 mg/kg. Recoveries obtained were in the range of 84 - 126 %, a detailed list of results is displayed below.

Matrix	Fort. Level (mg/kg)	% Recovery
Whole Plant	0.01	94
	0.02	84, 126
	0.04	104, 94, 109, 101
	0.6	95
Average:		101, s.dev. 13
Straw	0.02	108
	0.04	95, 91
	0.08	105, 99, 99
	0.6	85
Average:		97, s.dev. 8
Grain, Cob	0.02	96, 101
	0.04	96
	0.06	97
	0.3	97
Average:		97, s.dev. 2
Soil	0.01	97, 106, 97
	0.02	98, 117
	0.27	97
Average:		102, s.dev. 8
Total average		99.5 ± 8.8; n=26

The samples were taken from different corn residue trials performed at nine sites in France during 1989 and 1990. The soil samples were obtained from two sites in France which are representative for corn growing regions.

The recovery data obtained during the validation phase demonstrate that the method yields results with an accuracy required for residue analysis. Comparison of the average recoveries obtained for the four matrices proves that differences are not significant (95% C.I.).

We therefore conclude that the recovery of SAN 582 H does neither depend on the fortification level applied - and therefore not on the concentration of the analyte in the sample - nor on the matrix analyzed. The method therefore may be used as a routine method for the analysis of SAN 582 H in corn and soil samples.

SANDOZ AGRO LTD.
DEVELOPMENT REGION BASLE
ANALYTICAL CHEMISTRY AND ENVIRONMENTAL SCIENCES

TDS-No.: BS 1988

Table 1: Table of evaluated calibration data and limit of determination (first order function)

Limit of detection L1 : 0.029 $\mu\text{g/ml}$ (0.0035 mg/kg)
Limit of determination L2 : 0.044 $\mu\text{g/ml}$ (0.0053 mg/kg)

First Order Calibration Function

Method : SAN 582H in Corn
Detector : TSD
Active Ingredient : SAN 582H

```

*****
* c in  $\mu\text{g/ml}$       y in mm      sy in mm      n      *
*****
* 0.01            1.0           0.0           2      *
* 0.03            3.5           0.0           2      *
* 0.05            6.3           0.4           2      *
* 0.07            8.8           0.4           2      *
* 0.10            13.0          0.0           2      *
* 0.30            37.5          0.0           2      *
* 0.50            65.5          0.7           2      *
* 0.70            90.3          0.4           2      *
* 1.00            131.5         0.7           2      *
*****

```

c... concentration of standard solution
y... average detector response of n single injections
sy... s. dev. of n single injections
n... number of injections

Statistical Parameters

Intercept $a_0 = -0.54 \pm 0.34 \text{ mm}$
Slope $a_1 = 131.196 \pm 0.760 \text{ mm}/\mu\text{g/ml}$
No. of cal. levels $n = 9$
Degrees of freedom $fg = 7$
Stand. dev. of regr. $sy = 0.76 \text{ mm}$
Ratio (sa/a_1) $sy_0 = 5.79E-003 \mu\text{g/ml}$
Mean dev. for single det. $v = 1.889 \%$
Correlation coeff. $r = 0.999$
 t ($p=95\%$, fg) $t = 2.364$
Limit of detection $ng = 2.9E-002 \mu\text{g/ml}$
Limit of determination $bg = 4.4E-002 \mu\text{g/ml}$

SANDOZ AGRO LTD.
 DEVELOPMENT REGION BASLE
 ANALYTICAL CHEMISTRY AND ENVIRONMENTAL SCIENCES

TDS-No.: BS 1988

Table 2 : Table of evaluated calibration data and limit of detection (second order function)

Limit of detection L1 : 0.018 µg/ml (0.0022 mg/kg)
 Limit of determination L2 : 0.027 µg/ml (0.0032 mg/kg)

Second Order Calibration Function

Method : SAN 582H in Corn
 Detector : TSD
 Active Ingredient : SAN 582H

```

*****
* c in µg/ml   y in mm   sy in mm   n   *
*****
* 0.01         1.0       0.0        2   *
* 0.03         3.8       0.4        2   *
* 0.05         6.0       0.0        2   *
* 0.07         9.0       0.7        2   *
* 0.10        12.3       0.4        2   *
* 0.30        37.3       0.4        2   *
* 0.50        65.3       0.4        2   *
* 0.70        91.3       0.4        2   *
* 1.00       133.8       0.4        2   *
*****
    
```

c... concentration of standard solution
 y... average detector response of n single injections
 sy... s. dev. of n single injections
 n... number of injections

Statistical Parameters

Physical units : Standard : µg/ml
 Response : mm

Function : $y = a_0 + a_1x + a_2x^2$

Coefficients : $a_0 = -0.14 \text{ mm}$
 $a_1 = 124.28 \text{ mm/µg/ml}$
 $a_2 = 9.60 \text{ mm/(µg/ml}^2)$

Stand. dev. of regr. : $sy = 0.52 \text{ mm}$
 Sensitivity : $e = 130.17 \text{ mm/µg/ml}$
 Ratio (sy/a1) : $sx_0 = 0.0040 \text{ µg/ml}$
 Mean dev. for single det. : $v = 1.30 \%$
 Correlation coeff. : $r = 0.9998$
 Limit of detection : $ng = 1.8E-002 \text{ µg/ml}$
 Limit of determination : $bg = 2.7E-002 \text{ µg/ml}$

SANDOZ AGRO LTD.
DEVELOPMENT REGION BASLE
ANALYTICAL CHEMISTRY AND ENVIRONMENTAL SCIENCES

TDS-No.: BS 1988

Figure 1

DROPPING FUNNEL

EXTRELUT COLUMN

SILICA GEL COLUMN

ROUND BOTTOM FLASK

