Experiment 3: Practical Extractions/ Introduction to Statistical Methods

We will do Steps 1-5 first.

Steps 1 & 2 in pairs.

Procedure:

Students will work in pairs. Data generated by all students will be pooled for use in subsequent calculations to be submitted as part of the lab report.

1. A large bulk sample of contaminated sand will be provided. In pairs, cone and quarter the sand three times. Transfer the portion of sample selected onto wax paper. You will have about **60 g** of sample at this stage.
2. Table and then quarter the sample on the wax paper. At the end of this step you should have about **30 g** of sample (lab sample).

2x

1x

This is an unbiased method to reduce sample size for analysis.

We will do Steps 1-5 first.

Steps 3 onward – work individually

Tare a 250 mL Erlenmeyer flask and weigh 10 g contaminated sand in the flask.

Invert a small beaker to cover Erlenmeyer flask.

Work individually from this step onward.

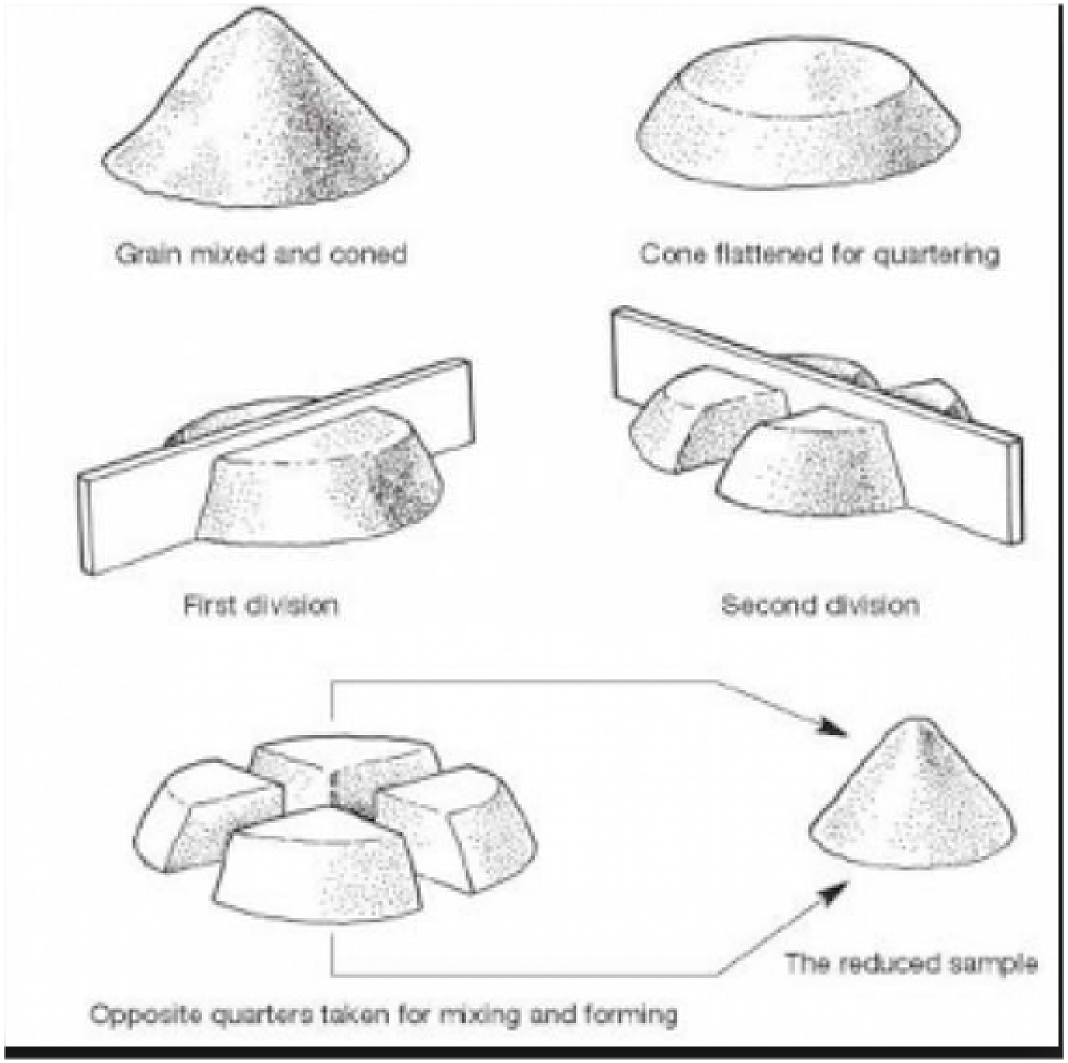
3. Clean the provided Erlenmeyer flask with acetone and then ensure that it is dry.
4. Weigh accurately 10 g of the contaminated sand sample in the flask.
5. In the fume hood, add 25 mL of dichloromethane, cover with a watch glass and then swirl gently. Place the flask on a magnetic stirrer and stir for about 60 to 70 minutes.

Wash bottle in the fume hood is dichloromethane.
No water is used today!!

Come back for lab lecture .

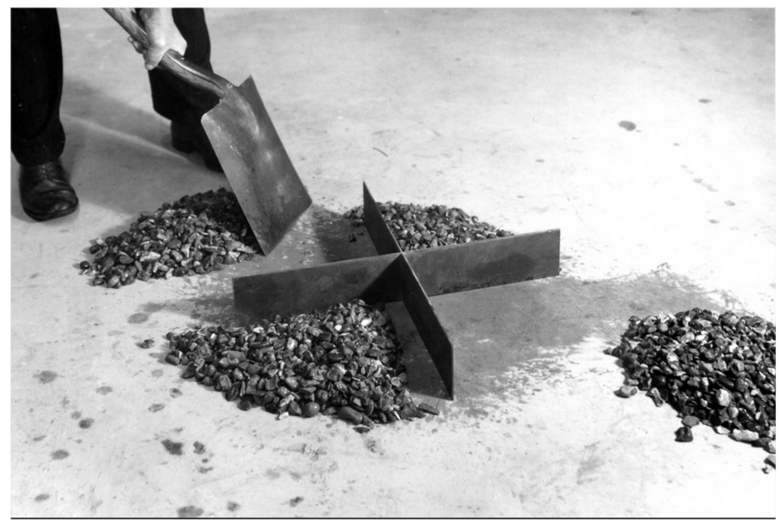
Stir the contaminated sand on LOW.
There should be NO sand splashed onto the side of the Erlenmeyer flask.

Demonstrate Cone and Quartering



This is to ensure that proper procedures are used to reduce sample size can lead to results that have significant biases and large imprecisions.

This is done on a large scale as well as on a tabletop scale.



Steps 3 onward – work individually

Wash the sand with small portions of dichloromethane while the sand is in the Erlenmeyer flask. It is not necessary to transfer all the sand into the filter paper.

Filter paper will NOT stick to the funnel.

6. In the fume hood, filter the sample using Whatman #1 filter paper which has been washed with a small amount of dicloromethane. Discard the washing.
7. Filter the contaminated sand mixture by gravity filtration. The filtrate should be collected in a **preweighed** round bottom flask that can be used with the rotary evaporator.
8. Rinse the Erlenmeyer flask with small portions of dichloromethane, transferring the washings to the filter. Finally rinse the filter paper with two 5 mL portions of dichloromethane.
9. With the aid of your instructor, remove the dichloromethane solvent using a rotary evaporator. Once all the solvent is removed, dry the outside of the flask and reweigh it. The weight of the extracted material is the difference between the two weighs.
10. The recovered dichloromethane solvent should be placed in a suitable container as it can be used again for subsequent extractions.

Put the round bottom flask on a tared weigh boat.

The round bottom flask should be at most half full.
DO NOT OVER RINSE!

Steps 3 onward – work individually

Due to extraneous matter.
0.030000 g per 10 g of sand

Theoretical value: 0.50000 g oil per 10 g sand.

11. Calculate the % oil in the sand. Remember to subtract the blank value provided by your instructor. The blank value is the weight of the dichloromethane extracted material if no oil has been added to the sand.
12. Assess the % recovery of the oil from the sand using the provided reference value for the amount of oil added to the sand.
13. Tabulate the % oil values for all the set. Calculate the mean, standard deviation and relative standard deviation of the results. Remember to test any suspect values using Dixon's Test.

Let's take a look at doing some statistics calculations on your calculator.

Outliers and the Dixon Q Test

Dixon Q Test: One of the methods used to test a set of results to see if any potential outliers can be identified.

Step 1: The data set containing N values is sorted either in an ascending or descending order. (E.g. $x_1, x_2, x_3, x_4, x_5, x_6, \ldots x_N$.)

Step 2: With x_1 being the suspected value, use the following equation to calculate Q.

$$Q = \frac{|x_1 - x_2|}{|x_1 - x_N|}$$

Step 3: Compare the value Q to the critical values listed in the table.

For a set of N results, if

$Q_{calc} > Q_{tabulated}$

then reject point.

Data is valid to 95% confident level

Q(P, N)			
N	P = 0.90*	P = 0.95**	P = 0.99***
3	0.89	0.94	0.99
4	0.68	0.76	0.89
5	0.56	0.64	0.76
6	0.48	0.56	0.70
7	0.43	0.51	0.64
8	0.40	0.47	0.58
9	-	0.44	-
10	-	0.41	-

Outliers and the Dixon Q Test

Analysis of percent sulfur in a coal sample: 4.01%, 3.99%, 3.96%, 4.13%. Use the Dixon Q Test table at 95% confident level to check if any of these data are outliers.

Step 1: The data set containing N values is sorted either in an ascending or descending order.
(E.g. $x_1, x_2, x_3, x_4, x_5, x_6, \ldots x_N$.)

3.96, 3.99, 4.01, 4.13

Test these data points.

$$Q = \frac{|x_1 - x_2|}{|x_1 - x_N|}$$

For a set of N results, if

$Q_{calc} > Q_{tabulated}$

then reject point.

Step 2: With x_1 being the suspected value, use the following equation to calculate Q.

Test 3.96: $$Q = \frac{3.99 - 3.96}{4.13 - 3.96} = \frac{0.03}{0.17} = 0.18$$

Test 4.13: $$Q = \frac{4.13 - 4.01}{4.13 - 3.96} = \frac{0.12}{0.17} = 0.71$$

Q(P, N)			
N	P = 0.90*	P = 0.95**	P = 0.99***
3	0.89	0.94	0.99
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9	-	0.44	-
10	-	0.41	-

Step 3: Compare the value Q to the critical values listed in the table.

In both cases, $Q_{calc} < Q_{tabulated}$, no outliers.

Outliers and the Dixon Q Test

Consider the following set of data:

Trials	#1	#2	#3	#4	#5	#6
Data (mg/L)	203	204	205	206	207	214

$$Q = \frac{|x_1 - x_2|}{|x_1 - x_N|}$$

Is the data point 214 mg/L in Trial #6 an outlier for a 95% confident level?

For a set of N results, if

$Q_{calc} > Q_{tabulated}$

then reject point.

Test 214: $$Q = \frac{214 - 207}{214 - 203} = \frac{7}{11} = 0.64$$

Since 0.64 > 0.56, this is an outlier.

Continue to test the other 5 points:

Test 207: $$Q = \frac{207 - 206}{207 - 203} = \frac{1}{4} = 0.25$$

Test 203: $$Q = \frac{204 - 203}{207 - 203} = \frac{1}{4} = 0.25$$

In both cases, $Q_{calc} < Q_{tabulated}$, no more outliers.

Q(P, N)			
N	P = 0.90*	P = 0.95**	P = 0.99***
3	0.89	0.94	0.99
4	0.68	0.76	0.89
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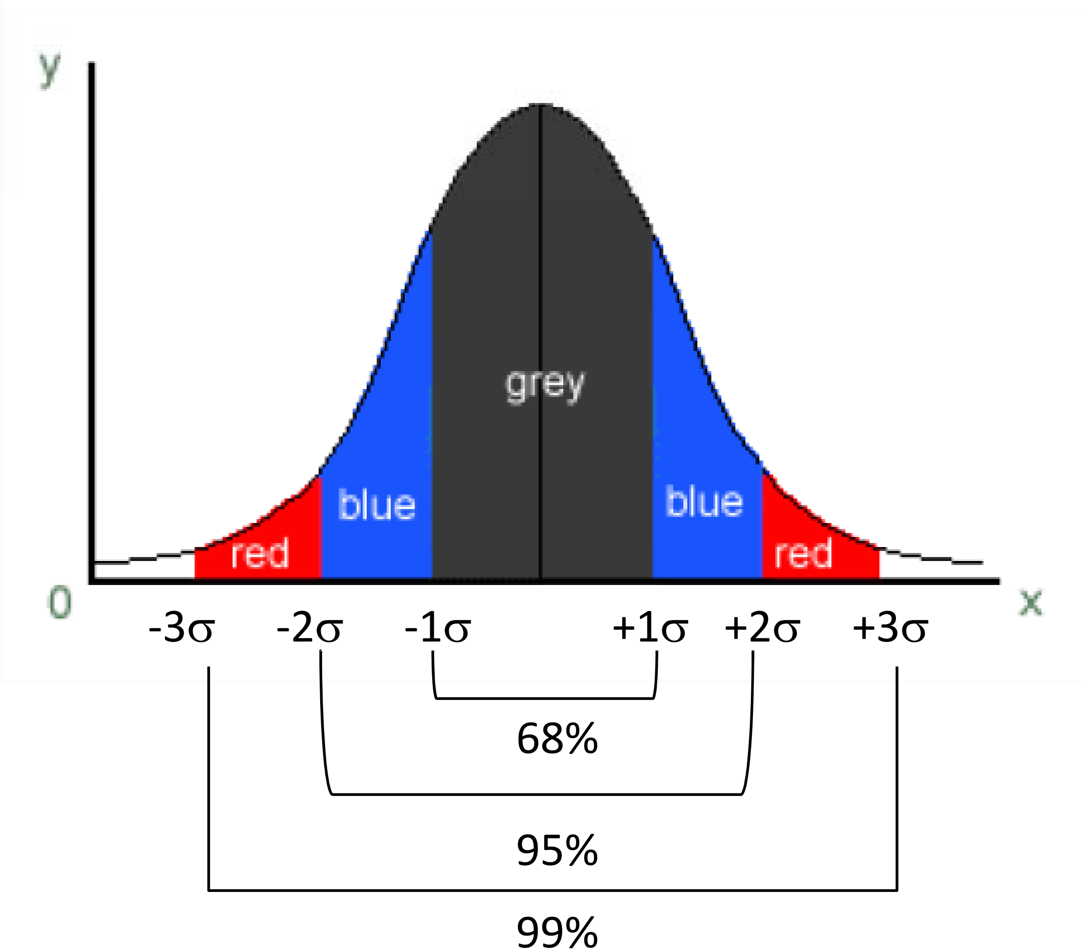
Statistics:

Standard deviation (SD) (represented by the Greek letter sigma, **σ**) tells how the different numbers in a data are scattered around the mean.

Relative SD (RSD) reflects spread of the data in percent. A higher relative standard deviation means that the numbers are widely spread from its average, while a lower relative standard deviation means the numbers are more closer to its average.



Sample SD, S

$$S = \sqrt{\frac{\sum_{i=1}^{N} (x_i - M)^2}{N - 1}}$$

N is the number of data values.
M is the mean.

$$M = \frac{x + y + z}{N}$$

$$RSD = \frac{100 \cdot S}{M}$$

Example:

An example of calculations of: (i) mean or average, (ii) standard deviation, and (iii) relative standard deviation.

Four measurements of lengths were taken: 51.3 cm, 55.6 cm, 49.9 cm and 52.0 cm.

$$M = \frac{x + y + z}{N}$$

(i) mean or average:

$$\text{mean} = \frac{51.3 + 55.6 + 49.9 + 52.0}{4} = \frac{208.8}{4} = 52.2 \text{ cm}$$

$$S = \sqrt{\frac{\sum_{i=1}^{N} (x_i - M)^2}{N - 1}}$$

i) standard deviation:

$$S = \sqrt{\frac{(51.3-52.2)^2 + (55.6-52.2)^2 + (49.9-52.2)^2 + (52.0-52.2)^2}{4 - 1}}$$

$$= \sqrt{\frac{(-0.9)^2 + (3.4)^2 + (-2.3)^2 + (-0.2)^2}{3}}$$

$$= \sqrt{\frac{0.81 + 11.56 + 5.29 + 0.04}{3}}$$

$$= \sqrt{5.9}$$

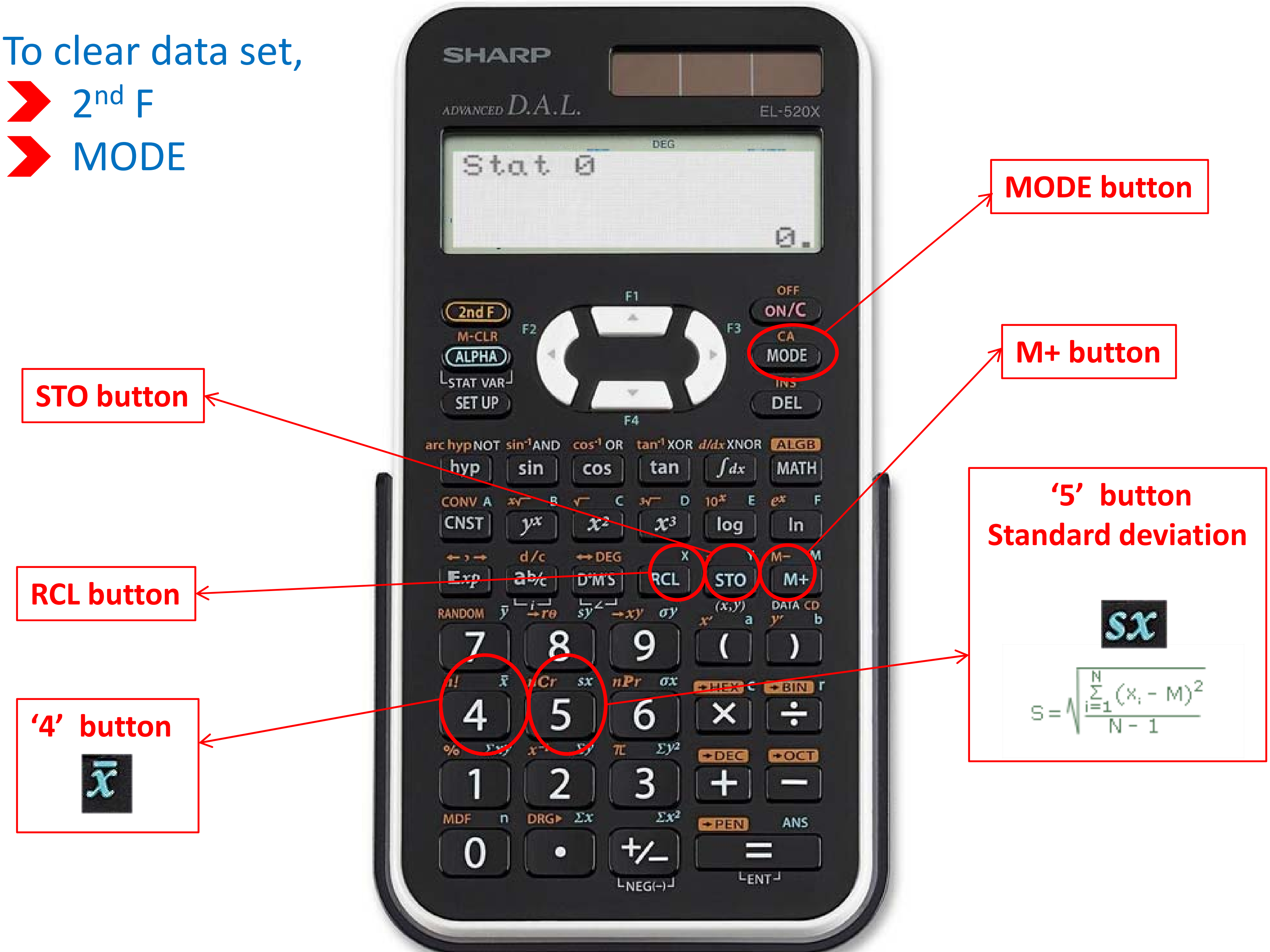
$$= 2.4$$

(iii) relative standard deviation, RSD = (S / mean) x 100 = 2.4 / 52.2 x 100 = 4.6 %

Therefore, for our measurement, we can write 52.2 ± 2.4 cm or 52.2 cm ± 4.6 %.

To clear data set,

- 2nd F
- MODE



Find M, SD, RSD using the calculator

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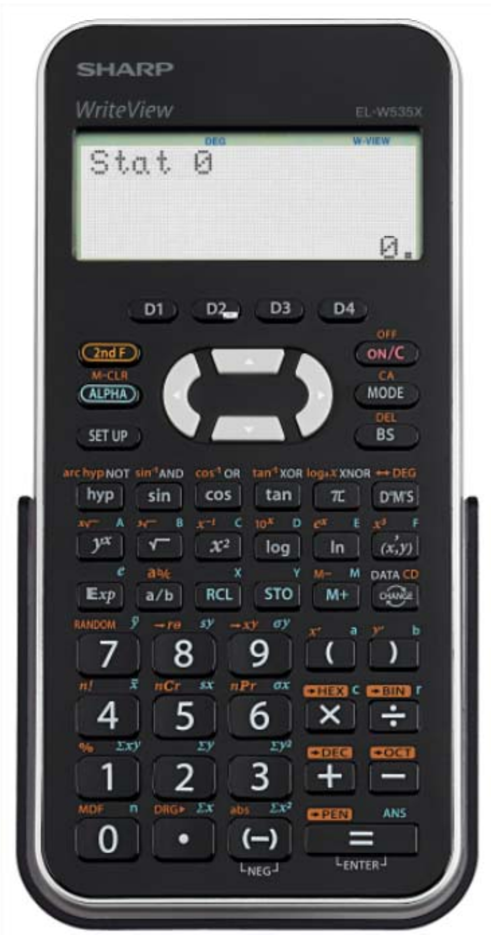

- MODE, 1, 0
- 51.3 DATA
- 55.6 DATA
- 49.9 DATA
- 52.0 DATA
- $\bar{x}$ RCL '4' = 52.2
- sx RCL '5' = 2.4
- RSD = ($^{\text{RCL '5'}}/_{\text{RCL '4'}}$) x 100 = 4.6

Find M, SD, RSD using the calculator

Data: 95, 80, 80, 75, 75, 75 , 50

- MODE, 1, 0
- 95 DATA
- 80 DATA
- DATA
- 75 STO 3 DATA
- 50 DATA
- $\bar{x}$ RCL ‘4’ — = 75.71428571 = 75.7
- sx RCL ‘5’ — = 13.3630621 = 13.4
- RSD = ($^{\text{RCL ‘5’}}/_{\text{RCL ‘4’}}$) x 100 — = 17.64919411 = 17.6%

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Test 203: $$Q = \frac{204 - 203}{207 - 203} = \frac{1}{4} = 0.25$$

No more outliers.

$\bar{x}$ = 205.0

sx = 1.6

RSD = (RCL '5' / RCL '4') x 100 = 0.77 %

Data Sheet:

1. Weight of sand (oven dried weight) (M) 9.9650 g ________________
(oil contaminated sand)

2. Weight of round bottom flask Don't forget to weigh. ________________

3. Weight of round bottom flask + extracted material ________________

4. Weight of extracted material (a) 0.4895 g ________________
(item 3 - item 2) (oil + extraneous matter)

Calculations:

Weight of extracted material for blank = 0.03000 g for 10 grams of material   Given.

Weight of extracted material for blank in Flask # =

0.030000 g ______ **x weight of sand/10 g =** 0.02990 g ______ (b)

9.9650 g

(from previous page)

1. % oil in the sand: (Show calculation in the space provided below. Remember to account for the blank.) Give this number to your instructor.

Weight of oil + extraneous matter

$$\% \text{ Oil} = \frac{\text{Weight of Oil Extracted}}{\text{Weight of Contaminated sand}} = \frac{(a) - (b)}{M} \cdot 100$$

$$\% \text{ Oil} = \frac{0.4895\text{g} - 0.02990\text{g}}{9.9650\text{g}} = \frac{0.4596\text{g}}{9.9650\text{g}} \cdot 100 = 4.612\%$$

Report your % oil in the sand on the board.

Calculations:

2. % recovery of the oil from the sand for Flask # ____________________
(Show calculation in the space provided below.)

Reference value for weight of oil spike = 0.5000 g. Given: $\frac{0.5000\text{g oil}}{10.000\text{g sand}}$

$$\text{Weight of oil} = \frac{0.5000\text{g oil}}{10.000\text{g sand}} \cdot 9.9650\text{g} = 0.4982\text{ g oil}$$

$$\%\ \text{Recovery} = \frac{\text{Weight of oil extracted}}{\text{Corrected Theoretical Value}} \cdot 100 = \frac{0.4596\text{g}}{0.4982\text{g}} \cdot 100 = 92.25\%$$

You will not get anywhere near 100% recovery.

Site Data:

Practical Extractions

Data Sheet:

Tabulate values for the whole set of results:

Site 1	Site 2	Site 3

Copy the data from the board for your site.

1. Perform Dixon Q test to test for outliers for data reported for your site.
2. Calculate mean, standard deviation and relative standard deviation for your site's data.