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Inter-Laboratory Comparison Measurements of Constituents of Oil Shale Standard Reference Sample

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Abstract

An inter-laboratory comparison study was carried out between twelve participating laboratories inside and outside Jordan using eleven different analytical methods. Meanwhile, the oil shale samples sent to each participant laboratory and asked for analysis using the available analytical technique/s. The oil shale sample was characterized for the following parameters: oil content, total carbon, organic carbon, ash, calorific value, moisture, loss on ignition, minerals, oxides, major and minor elements in twelve participating laboratories inside and outside Jordan using eleven different analytical methods. Meanwhile, the oil shale samples sent to each participant laboratory and asked for analysis using the available analytical technique/s. The results collected from participant laboratories were analyzed statistically after outlying the extreme values applying Q-test and evaluated applying z scores for satisfying the result of each participant.

Keywords: SJL-1; Inter-laboratory comparison; Oil shale; El-lujjon

1. Introduction

The University of Jordan has organized the First national inter-laboratory Proficiency Testing Scheme (PTS), in cooperation with the Prince Faisal Center for Dead Sea Environmental and Energy Researches, Mutah University, Jordan for analysis of oil shale standard reference sample by participating laboratories from Jordan and from other countries.

The intra-laboratory reproducibility will be used in the inter-laboratory comparison between different contributing laboratories. The results of this study will be used for determining the performance of individual laboratories for each measurement and to monitor laboratories continuing performance, detect problems in laboratories such as staff performance or calibration of instruments. Then based on these results an action can be taken to monitor and compare between the established methods of measurements, laboratories confidence of clients increased. Finally, the main aim of the interlaboratory

Comparison is to demonstrate competence and to establish degree of agreement between results of the participating laboratories that leads to assign certified value for reference materials (1, 2, 6, 7, 8).

2. Methods and Statistical evaluation

Oil shale sample SJL-1 was sent and analyzed in different participating laboratories with different analysis methods as in Table (38).

Statistical evaluation is performed by the organizer who collects the results from the participant laboratories and compiles in report and utilizes software to check for agreement with the definitions.

The concept of Z scores has also been used for evaluation of laboratories performance. The value of Z is estimated usually according to the following equation:

 $|\mathbf{Z}| = (\mathbf{Xi} - \overline{X})/\mathbf{S}$ Equation (1)

Where

Xi: the average value measured in each laboratory for each constituent

X: mean value for average values collected from each laboratory for each constituent **S**: standard deviation.

The following criteria are applied for the final evaluation

- $|Z| \le 2$ satisfactory
- 2 < |Z| < 3 questionable
 - |Z| > 3 unsatisfactory

Z value considered as the most popular parameter applied in the inter laboratory comparison evaluation. (3, 4, 5). The contributing laboratories in analysis and certification process should analyze the sample with high level of care as a special sample not as a routine one. The participating laboratories were requested to identify the methods used for measuring each parameter in submitted reports according to the analytical method codes, several replicates or repetitions should be carried out by each laboratory for sample analysis to check for reproducibility, the graphical plot shows the lower and upper boundaries as Z values, the zero line is the fit with

mean value (1, 3).

3. Results and Discussion

The scheme which complies with the requirements of the ISO/IEC Guide 43:1997 was organized among 12 national and international accredited participating laboratories (3, 4).

The parameters to be tested in the scheme were chosen to represent the types of analysis generally analyzed in participating laboratories oil shale samples. The inter-laboratory comparison between participant laboratories was performed by applying z score and estimating the z value for each laboratory with each method according to Equation 1. The results are presented in Tables (1-37) show z scores for participant laboratories in analysis. Most compared constituents of oil shale sample SJL-1 analyzed in different laboratories have absolute value of $|z| \le 2$ after last rejection. This means that it is satisfactory according to ISO 5725. Except of some parameters such as Al₂O₃ at RSSJ, XRF that has |z| = 2.16 and TiO₂ with |z| = 2.1. Also Fe₂O₃ total has |z| = 2.148 at IECTUB XRF. **Table (1):** Aluminum Oxide (Al₂O₃) in % (wt/wt)

Lab code	Analytical Method Code	Reported Value	z-score	Rating
106	WDXRF	2.930	2.160	Questionable
108	WDXRF	2.230	-1.440	Satisfactory
101	WDXRF	2.500	-0.051	Satisfactory
101	ICP/MS	2.538	0.154	Satisfactory
110	WDXRF	2.530	0.103	Satisfactory
110	AAS	2.500	-0.051	Satisfactory
110	EDXRF	2.540	-0.154	Satisfactory
102	ICP/OES	2.620	0.566	Satisfactory
102	ICP/OES	2.450	-0.309	Satisfactory
103	WDXRF	2.604	0.463	Satisfactory
111	WDXRF	2.201	-1.596	Satisfactory

Number of Reported Values = 11 Data Range 2.930-2.201 Assigned Value = 2.513 Standard Deviation = 0.194

Table (2): Phosphorous Oxide (P2O5) in % (wt/wt)

Lab code	Analytical Method Code	Reported Value	z-score	Rating
106	WDXRF	2.913	0.1406	Satisfactory
108	WDXRF	2.830	-0.6928	Satisfactory
101	WDXRF	2.730	-1.697	Satisfactory
101	ICP/MS	3.017	1.185	Satisfactory
110	EDXRF	2.990	0.914	Satisfactory
103	WDXRF	2.864	-0.351	Satisfactory
111	WDXRF	2.950	0.512	Satisfactory

Number of Reported Values = 7 Data Range 3.017-2.730 Assigned Value = 2.899 Standard Deviation = 0.010

Table (3) : Manganese Oxide (MnO) in %	(wt/wt)	
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Lab code	Analytical Method Code	Reported Value	z-score	Rating
105	ICP/MS	0.0042	1.182	Satisfactory
101	ICP/MS	0.0037	0.118	Satisfactory
110	WDXRF	0.0033	-0.922	Satisfactory
102	ICP/OES	0.0040	0.733	Satisfactory
111	WDXRF	0.0032	-1.097	Satisfactory

Number of Reported Values = 5

Data Range 0.0042-0.0032

Assigned Value = 0.0037 Standard Deviation = 0.0004

Table (4): Calcium Oxide (CaO) in % (wt/wt)

Lab code	Analytical Method Code	Reported Value	z-score	Rating
106	WDXRF	26.440	0.391	Satisfactory
108	WDXRF	24.000	637	Satisfactory
101	WDXRF	24.870	-0.271	Satisfactory
109	AAS	22.730	-1.172	Satisfactory
101	NA	23.320	0924	Satisfactory
101	ICP/MS	23.350	-0.911	Satisfactory
110	WDXRF	26.580	0.450	Satisfactory
110	EDXRF	24.900	-0.263	Satisfactory
102	ICP/OES	29.700	1.765	Satisfactory
102	ICP/OES	25.900	0.163	Satisfactory
103	EDXRF	30.610	2.148	Questionable
103	WDXRF	24.454	-0.446	Satisfactory
111	WDXRF	24.800	-0.712	Satisfactory

Number of Reported Values = 13

Data Range 30.610-22.730

Assigned Value = 25.512

Standard Deviation = 2.373

Table (5): Titanium Oxide (TiO_2) in % (wt/wt)

Lab code	Analytical Method Code	Reported Value	z-score	Rating
106	WDXRF	0.174	2.015	Questionable
108	WDXRF	0.110	-1.206	Satisfactory
101	WDXRF	0.123	-0.550	Satisfactory
109	AAS	0.152	0.889	Satisfactory
101	ICP/MS	0.127	-0.335	Satisfactory
110	EDXRF	0.115	-0.955	Satisfactory
110	WDXRF	0.116	-0.904	Satisfactory
102	ICP/OES	0.134	0.00252	Satisfactory
102	ICP/OES	0.144	0.5063	Satisfactory
103	WDXRF	0.145	0.5345	Satisfactory

Number of Reported Values = 10 Data Range 0.174-0.110 Assigned Value = 0.134 Standard Deviation = 0.020

 Table (6):
 Magnesium Oxide (MgO) in % (wt/wt)

Lab code	Analytical Method Code	Reported Value	z-score	Rating
106	WDXRF	0.630	-0.832	Satisfactory
109	AAS	0.675	-0.153	Satisfactory
101	ICP/MS	0.668	-0.2496	Satisfactory
110	EDXRF	0.585	-1.401	Satisfactory
110	WDXRF	0.745	0.818	Satisfactory
102	ICP/OES	0.775	1.234	Satisfactory
103	WDXRF	0.778	1.276	Satisfactory
111	WDXRF	0.634	-0.757	Satisfactory

Number of Reported Values = 8 Data Range 0.778-0.585

Assigned Value = 0.686

Standard Deviation = 0.072

Table (7): Potassium Oxide (K₂O) in % (wt/wt)

Lab code	Analytical Method Code	Reported Value	z-score	Rating
106	WDXRF	0.313	-1.667	Satisfactory
108	WDXRF	0.330	-1.062	Satisfactory
105	ICP/MS	0.339	-0.741	Satisfactory
101	ICP/MS	0.374	0.506	Satisfactory
110	WDXRF	0.393	1.183	Satisfactory
110	EDXRF	0.366	0.2209	Satisfactory
102	ICP/OES	0.365	0.185	Satisfactory
102	ICP/OES	0.351	-0.313	Satisfactory
103	WDXRF	0.406	1.646	Satisfactory
111	WDXRF	0.361	0.043	Satisfactory

Number of Reported Values = 10

Data Range 0.406-0.313

Assigned Value = 0.360

Standard Deviation = 0.028

Table (8): Ferric Oxide (Fe₂O₃T) in % (wt/wt)

Lab code	Analytical Method Code	Reported Value	z-score	Rating
106	WDXRF	0.737	-1.664	Satisfactory
108	WDXRF	1.010	0.310	Satisfactory
101	WDXRF	1.027	0.433	Satisfactory
109	AAS	0.943	-0.174	Satisfactory
105	ICP/MS	0.744	-1.613	Satisfactory
101	ICP/MS	1.025	0.419	Satisfactory
110	WDXRF	0.987	0.144	Satisfactory
110	EDXRF	0.940	-0.196	Satisfactory
110	AAS	0.940	-0.196	Satisfactory
103	WDXRF	0.944	-0.167	Satisfactory
103	EDXRF	1.268	2.175	Questionable
111	WDXRF	1.041	0.534	Satisfactory

Number of Reported Values = 12 Data Range 1.268-0.737 Assigned Value 0.967 Standard Deviation = 0.138

 Table (9): Sodium Oxide (Na₂O) in % (wt/wt)

Lab code	Analytical Method Code	Reported Value	z-score	Rating
106	WDXRF	0.182	0.1936	Satisfactory
108	WDXRF	0.190	0.581	Satisfactory
101	WDXRF	0.153	-1.210	Satisfactory
109	AAS	0.148	-1.452	Satisfactory
101	NA	0.162	-0.774	Satisfactory
101	ICP/MS	0.188	0.484	Satisfactory
110	WDXRF	0.205	1.307	Satisfactory
110	EDXRF	0.209	1.500	Satisfactory
102	ICP/OES	0.168	-0.484	Satisfactory
102	ICP/OES	0.175	-0.145	Satisfactory

Number of Reported Values = 10

Data Range 0.209-0.148 Assigned Value = 0.178

Standard Deviation = 0.021

Table (10): Uranium (U) in ppm

Lab code	Analytical Method Code	Reported Value	z-scores	Rating
111	ICP/MS	28.206	0.270	Satisfactory
105	ICP/MS	22.330	-1.407	Satisfactory
111	Gamma	28.760	0.428	Satisfactory
101	NA	26.670	-0.169	Satisfactory
101	ICP/MS	32.600	1.523	Satisfactory
110	EDXRF	25.000	-0.645	Satisfactory

Number of Reported Values = 6 Data Range 32.600-22.330 Assigned Value = 27.261 Standard Deviation = 3.505

Table (11): Zinc (Zn) in ppm

Lab code	Analytical Method Code	Reported Value	z-score	Rating
109	AAS	665.04	-1.044	Satisfactory
105	ICP/MS	756.61	0.0138	Satisfactory
106	ICP/OES	661.87	-1.080	Satisfactory
101	NA	740.00	-0.178	Satisfactory
101	ICP/MS	759.33	0.045	Satisfactory
110	WDXRF	740.00	-0.178	Satisfactory
110	EDXRF	660.00	-1.102	Satisfactory
102	ICP/OES	938.00	2.108	Questionable
103	EDXRF	808.33	0.611	Satisfactory
111	WDXRF	825.00	0.804	Satisfactory

Number of Reported Values = 10

Data Range 938.00-660.00

Assigned Value = 755.42

Standard Deviation = 86.60

Table (12): Vanadium (V) in ppm

Lab code	Analytical Method Code	Reported Value	z-score	Rating
109	AAS	261.33	-0.303	Satisfactory
105	ICP/MS	261.59	-0.283	Satisfactory
101	ICP/MS	248.33	-1.301	Satisfactory
110	WDXRF	269.00	0.285	Satisfactory
110	EDXRF	260.00	-0.405	Satisfactory
102	ICP/OES	289.00	1.820	Satisfactory
102	ICP/OES	278.00	0.976	Satisfactory
111	WDXRF	255.00	-0.789	Satisfactory

Number of Reported Values = 8 Data Range 289.00-248.33 Assigned Value = 265.28

Standard Deviation = 13.03

Table (13): Strontium (Sr) in ppm

Lab code	Analytical Method Code	Reported Value	z-score	Rating
109	AAS	207.57	0.745	Satisfactory
106	ICP/OES	162.70	-1.650	Satisfactory
101	NA	196.67	0.164	Satisfactory
101	ICP/MS	189.67	-0.210	Satisfactory
110	WDXRF	188.00	-0.299	Satisfactory
111	ICP/MS	217.02	1.250	Satisfactory

Number of Reported Values = 9

Data Range 923.667-670.000

Assigned Value = 800.519

Standard Deviation = 84.662

Table (14): Nicel (Ni) in ppm

Lab code	Analytical Method Code	Reported Value	z-score	Rating
106	ICP/OES	731.130	-0.820	Satisfactory
101	ICP/MS	832.330	0.376	Satisfactory
110	EDXRF	690.000	-1.305	Satisfactory
110	WDXRF	670.000	-1.542	Satisfactory
111	ICP/MS	861.545	0.721	Satisfactory
102	ICP/OES	821.000	0.242	Satisfactory
102	ICP/OES	827.000	0.313	Satisfactory
103	EDXRF	923.667	1.455	Satisfactory
111	WDXRF	848.000	0.561	Satisfactory

Number of Reported Values = 6 Data Range 217.02-162.70 Assigned Value = 193.61 Standard Deviation = 18.73

Table (15): Sulfur (S) in ppm

Lab code	Analytical Method Code	Reported Value	z-score	Rating
107	ELEMEN	3.987	0.649	Satisfactory
108	ELEMEN	3.435	-0.377	Satisfactory
101	ELEMEN	3.480	-0.294	Satisfactory
101	ICP/MS	3.330	-0.572	Satisfactory
110	EDXRF	4.540	1.676	Satisfactory
102	ICP/OES	2.930	-1.316	Satisfactory
102	ICP/OES	4.160	0.970	Satisfactory
103	ELEMEN	3.242	-0.736	Satisfactory

Number of Reported Values = 8

Data Range 4.540-2.930

Assigned Value = 3.638 Standard Deviation = 0.5387

 Table (16):
 Cupper (Cu) in ppm

Lab code	Analytical Method Code	Reported Value	z-score	Rating
109	AAS	104.70	1.009	Satisfactory
105	ICP/MS	98.65	0.207	Satisfactory
101	ICP/MS	103.47	0.846	Satisfactory
110	WDXRF	86.00	-1.469	Satisfactory
102	ICP/OES	89.80	-0.966	Satisfactory
102	ICP/OES	104.00	0.916	Satisfactory
103	EDXRF	93.00	-0.542	Satisfactory

Number of Reported Values = 7

Data Range 104.70- 86.00

Assigned Value = 97.09 Standard Deviation = 7.55

Table (17): Chromium (Cr) in ppm

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Lab code	Analytical Method Code	Reported Value	z-score	Rating	
109	AAS	347.610	-1.442	Satisfactory	
105	ICP/MS	412.620	0.479	Satisfactory	
106	ICP/OES	347.530	-1.445	Satisfactory	
101	NA	396.670	0.008	Satisfactory	
101	ICP/MS	415.330	0.560	Satisfactory	
110	WDXRF	395.000	-0.041	Satisfactory	
110	EDXRF	411.000	0.432	Satisfactory	
111	ICP/MS	445,435	1.450	Satisfactory	

Number of Reported Values = 8

Data Range 445.435-347.530

Assigned Value = 396.399

Standard Deviation = 33.827

Table (18): Silicon Oxide(SiO₂) in % (wt/wt)

Lab code	Analytical Method Code	Reported Value	z-score	Rating
106	WDXRF	27.280	1.252	Satisfactory
108	WDXRF	25.300	-0.967	Satisfactory
101	WDXRF	26.250	0.097	Satisfactory
110	WDXRF	26.850	0.770	Satisfactory
110	AAS	24.810	-1.517	Satisfactory
103	WDXRF	26.782	0.694	Satisfactory
111	WDXRF	25.870	-0.328	Satisfactory

Number of Reported Values = 7 Data Range 27.280-24.810 Assigned Value = 26.163 Standard Deviation = 0.892

 Table (19): Molybdenum (Mo) in ppm

Lab code	Analytical Method Code	Reported Value	z-score	Rating
109	AAS	259.05	1.521	Satisfactory
101	NA	195.33	-0.042	Satisfactory
101	ICP/MS	145.93	-1.254	Satisfactory
102	ICP/OES	184.00	-0.320	Satisfactory
102	ICP/OES	201.00	0.097	Satisfactory

Number of Reported Values = 5 Data Range 259.05-145.93 Assigned Value = 197.06 Standard Deviation = 40.76

Table (20): Lead (Pb) in ppm

Lab code	Analytical Method Code	Reported Value	z-score	Rating
109	AAS	44.350	1.489	Satisfactory
105	ICP/MS	4.495	-1.197	Satisfactory
106	ICP/MS	25.380	0.210	Satisfactory
101	ICP/MS	5.270	-1.145	Satisfactory
111	ICP/MS	9.927	-0.831	Satisfactory
102	ICP/OES	34.305	0.812	Satisfactory
102	ICP/OES	34.000	0.791	Satisfactory
103	EDXRF	44.350	-0.1299	Satisfactory

Number of Reported Values = 7

Data Range 44.350-4.495

Assigned Value = 22.257

Standard Deviation = 14.836

 Table (21): Loss On Ignition (LOI) in %(wt/wt)

Lab code	Analytical Method Code	Reported Value	z-score	Rating
106	GRAV	36.92	-0.095	Satisfactory
108	GRAV	35.80	-1.245	Satisfactory
101	GRAV	38.03	1.044	Satisfactory
109	GRAV	35.91	-1.132	Satisfactory
110	GRAV	37.57	0.572	Satisfactory
111	GRAV	37.85	0.859	Satisfactory

Number of Reported Values = 6

Data Range 69.00-41.83 Assigned Value = 56.33 Standard Deviation = 11.10

Table (22): Barium(Ba) in ppm

Lab code	Analytical Method Code	Reported Value	z-score	Rating
106	ICP/OES	41.83	-1.307	Satisfactory
101	NA	50.00	-0.571	Satisfactory
101	ICP/MS	65.33	0.811	Satisfactory
102	ICP/OES	55.50	-0.075	Satisfactory
102	ICP/OES	69.00	0.020	Satisfactory

Number of Reported Values = 5 Data Range 41.83-27.17 Assigned Value = 56.33 Standard Deviation = 11.10

Table (23): Lithium (Li) in ppm

Lab code	Analytical Method Code	Reported Value	z-score	Rating	
105	ICP/MS	9.095	0.709	Satisfactory	
101	ICP/MS	7.630	-0.705	Satisfactory	

Number of Reported Values = 2
Data Range 9.095-7.630
Assigned Value = 8.362
Standard Dariation - 1 020

Standard Deviation = 1.036

Table (24): Ash in %(wt/wt)

Lab code	Analytical Method Code	Reported Value	z-score	Rating
109	GRAV	63.97	0.669	Satisfactory
106	GRAV	63.08	-0.416	Satisfactory
108	GRAV	64.20	0.953	Satisfactory
110	GRAV	62.43	-1.210	Satisfactory

Number of Reported Values = 4 Data Range 64.20-62.43 Assigned Value = 63.42 Standard Deviation = 0.82

Table (25): Cadmium (Cd) in ppm **Analytical Method Code Reported Value** Lab code z-score Rating -0.240 **ICP/OES** 64.57 Satisfactory 106 101 ICP/MS 78.17 0.445 Satisfactory 110 EDXRF 37.00 -1.629 Satisfactory ICP/OES 102 79.50 0.512 Satisfactory **ICP/OES** 87.40 0.910 102 Satisfactory

Number of Reported Values = 5 Data Range 87.40-37.00

Assigned Value = 69.33

Standard Deviation = 19.85

Table (26): Astatine (As) in ppm

Lab code	Analytical Method Code	Reported Value	z-score	Rating
105	ICP/MS	19.875	1.284	Satisfactory
101	NA	15.670	0.297	Satisfactory
102	ICP/OES	11.400	-0.704	Satisfactory
103	EDXRF	10.667	-0.876	Satisfactory

Number of Reported Values = 4 Data Range 19.875-10.667 Assigned Value = 14.403 Standard Deviation = 4.263

Table (27): Hydrogen(H) in %(wt/wt)

Lab code	Analytical Method Code	Reported Value	z-score	Rating
107	ELEMEN	2.122	-0.707	Satisfactory
108	ELEMEN	2.348	0.707	Satisfactory

Number of Reported Values = 2

Data Range 2.348-2.122 Assigned Value = 2.235

Standard Deviation = 0.160

Table (28): Nitrogen(N) in %(wt/wt)

Lab code	Analytical Method Code	Reported Value	z-score	Rating
107	ELEMEN	0.2610	-0.495	Satisfactory
108	ELEMEN	0.9050	1.420	Satisfactory
103	ELEMEN	0.4185	-0.050	Satisfactory
103	ELEMEN	0.1565	-0.841	Satisfactory

Number of Reported Values = 4 Data Range 0.9050-0.1565 Assigned Value = 0.4352 Standard Deviation = 0.3312

Table (29): Rubidium(Rb) in ppm

Lab code	Analytical Method Code	Reported Value	z-score	Rating
105	ICP/MS	9.225	-0.815	Satisfactory
101	NA	15.000	1.439	Satisfactory
101	ICP/MS	10.030	-0.501	Satisfactory
103	EDXRF	11.000	-0.123	Satisfactory

Number of Reported Values = 4

Data Range 3.030-2.770

Assigned Value = 2.900

Standard Deviation = 0.184

Table (30): Thorium(Th) in ppm

Lab code	Analytical Method Code	Reported Value	z-score	Rating
101	NA	1.27	-0.962	Satisfactory
101	ICP/MS	1.60	1.034	Satisfactory
111	ICP/MS	1.42	-0.071	Satisfactory

Number of Reported Values = 3

Data Range 1.60-1.27

Assigned Value = 1.43

Standard Deviation = 0.16

Table (31): Yorinum (Y) in ppm

Lab code	Analytical Method Code	Reported Value	z-score	Rating
101	ICP/MS	22.2	-0.176	Satisfactory
110	EDXRF	20.0	-0.900	Satisfactory
103	EDXRF	26.0	1.076	Satisfactory

Number of Reported Values = 3

Data Range 26.0-20.0

Assigned Value = 22.7

Standard Deviation = 3.0

 Table (32):
 Zirconium(Zr) in ppm

Lab code	Analytical Method Code	Reported Value	z-score	Rating
101	ICP/MS	18.53	-1.142	Satisfactory
110	EDXRF	31.00	0.720	Satisfactory
110	WDXRF	29.00	0.421	Satisfactory

Number of Reported Values = 3

Data Range 31.00-18.53

Assigned Value = 26.18 Standard Deviation = 6.70

Table (33): Selenium(Se) in ppm

Lab code	Analytical Method Code	Reported Value	z-score	Rating
101	NA	39.67	-0.707	Satisfactory
110	EDXRF	44.00	0.707	Satisfactory

Number of Reported Values = 2

Data Range 44.00-39.67 Assigned Value = 41.83

Standard Deviation = 3.06

Table (34): Total Carbon (C) in ppm

Lab code	Analytical Method Code	Reported Value	z-score	Rating
107	ELEMEN	20.740	-0.665	Satisfactory
108	ELEMEN	23.462	1.462	Satisfactory
101	ELEMEN	21.370	-0.173	Satisfactory
103	ELEMEN	20.791	-0.625	Satisfactory

Number of Reported Values = 4 Data Range 23.462-20.740 Assigned Value = 21.591 Standard Deviation = 1.280

Table (35): Moisture (H₂O) in %(wt/wt)

Lab code	Analytical Method Code	Reported Value	z-score	Rating
109	GRAV	0.680	-0.613	Satisfactory
106	GRAV	0.702	-0.542	Satisfactory
103	GRAV	1.228	1.155	Satisfactory

Number of Reported Values = 3 Data Range 1.228-0.680 Assigned Value = 0.870 Standard Deviation = 0.310

Table (36): Cobalt (Co) in ppm

Lab code	Analytical Method Code	Reported Value	z-score	Rating
105	ICP/MS	2.940	0.569	Satisfactory
101	NA	2.670	0.089	Satisfactory
101	ICP/MS	2.670	0.089	Satisfactory
111	ICP/MS	3.148	0.939	Satisfactory
102	ICP/OES	1.683	-1.667	Satisfactory

Number of Reported Values = 5

Data Range 3.148-1.683

Assigned Value = 2.622

Standard Deviation = 0.562

Table (37): Organic carbon(C org) in %(wt/wt)

Lab code	Analytical Method Code	Reported Value	z-score	Rating
108	ELEMEN	16.50	0.307	Satisfactory
103	ELEMEN	14.89	-1.117	Satisfactory
103	ELEMEN	17.07	0.811	Satisfactory

Number of Reported Values = 3

Data Range 17.07-14.89

Assigned Value = 16.15

Standard Deviation = 1.13

Table (38): Analytical Methode Codes

Analytical methods	Analytical Method abbreviation	
Inductively Coupled Plasma Mass Spectrometry	ICP/MS	
Inductively Coupled Plasma Optical Emission Spectroscopy	ICP/OES	
Atomic Absorption Spectroscopy	AAS	
Neutron Activation	NA	
Elemental analysis	ELEMEN	
Gravimetric	GRAV	
Energy Dispersive X-Ray Fluorescence	EDXRF	
Wave Length Dispersive X-Ray Fluorescence	WDXRF	
Gamma Spectrometry	Gamma	

4. Conclusion

Participation of laboratories in inter-laboratory comparisons considered one of the most important external tools of quality control. Each participating laboratory will received evaluation report with code number in order to compare its results with the results of other laboratories, determine the errors and take action. Generally, it is indicated that most results analyzed after last rejection from different laboratories are acceptable. In the future we hope to do more inter-laboratory comparisons with more laboratories and new methods and techniques to get certified values of the samples constituents.

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- Natural Resources Authority Laboratories, Amman -Jordan.
- Aqaba Special Economic Zone Authority BEN HAYYAN-Aqaba International Laboratories, Aqaba-Jordan.
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- Jordan Atomic Energy Commission Laboratories, Amman-Jordan.
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