7th Workshop

PROFICIENCY TESTING IN ANALYTICAL CHEMISTRY, MICROBIOLOGY AND LABORATORY MEDICINE

Current Practice and Future Directions

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A Proficiency Testing Scheme for Fuel Analysis Laboratories in Turkey: 'LABKAR'

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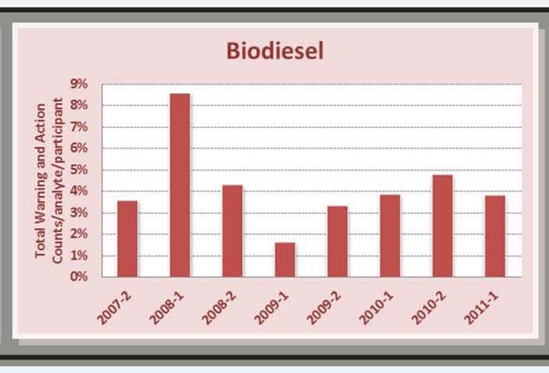
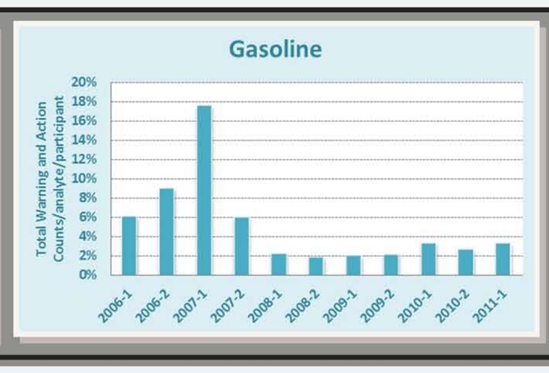
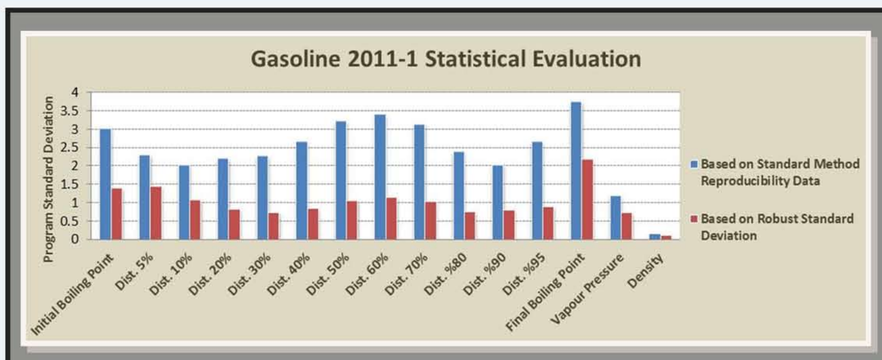
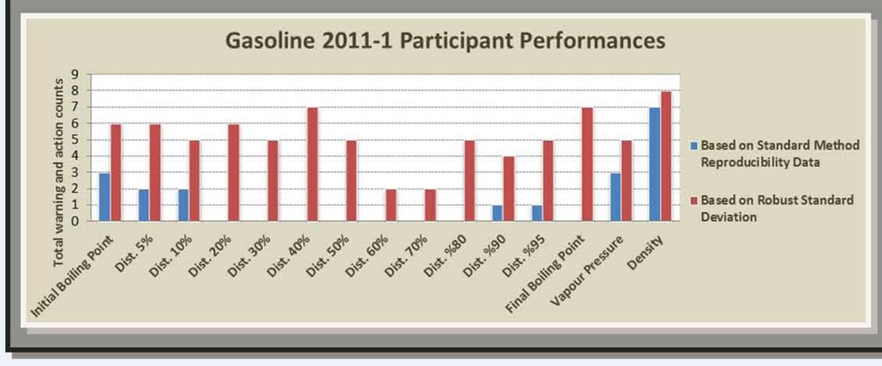
LABKAR is the name of the working group which was established to provide proficiency testing (PT) schemes for the fuel analysis laboratories in Turkey. The proficiency testing operations are being carried out by Middle East Technical University Petroleum Research Center (PAL) in Ankara. The center is performing quality control tests of the fuel products as well as research and development projects for the sector since 1991.



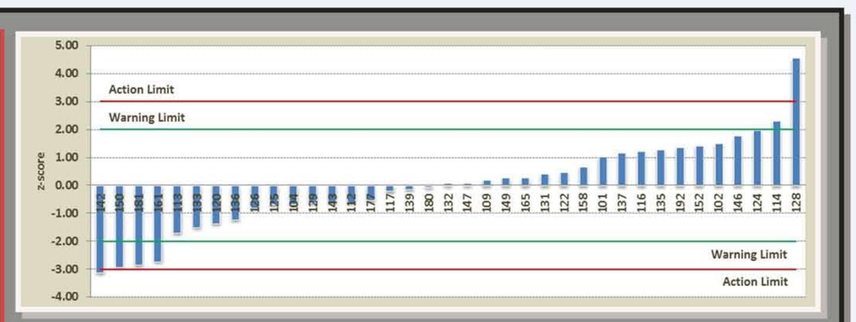


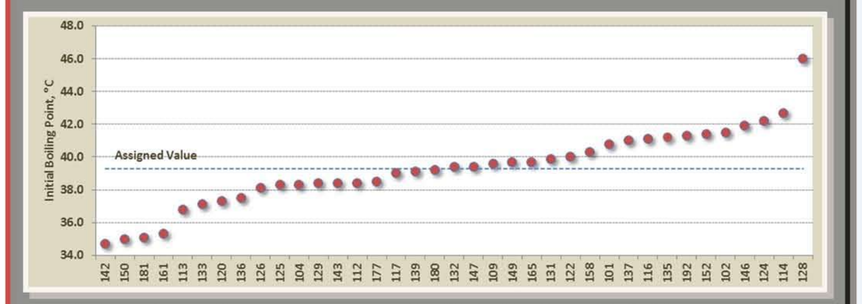
The effect of the scheme on the performance of the participants can be seen in the figures left. As seen, total warning and action counts per analyte per participant values have decreased in time except the initial phase of the scheme. The reason of this delay can be due to a late action from the participants and the addition of extra analytes.



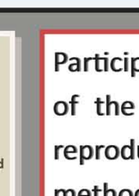
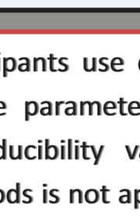


Participants use different methods in measurement of the parameters which means using a constant reproducibility value coming from one of these methods is not appropriate. Therefore it was decided to change the program standard deviation calculation methodology. Robust standard deviation value is used instead of standard methods' reproducibility value in calculation of program standard deviation. This change will result in a decrease in the program standard deviation values. The effect is shown in the figures for first Gasoline cycle performed in 2011. Total warning and action counts is also increased. Another outcome of using robust standard deviation is to be able to evaluate the analytes which have no reproducibility values.





Participant performances on 'Initial Boiling Point' analyte for gasoline 2011 first cycle.

Parameter	Gasoil	Gasoline	LPG	Biodiesel	Mineral Oil	Fuel Oil
						
Distillation	✓	✓	×	×	×	×
Setan Index	✓	×	×	×	×	×
Density	✓	✓	×	✓	✓	✓
Sulphur Content	✓	✓	✓	✓	×	✓
Total Contamination	✓	×	×	✓	×	×
Water Content	✓	×	×	✓	×	✓
CFPP	✓	×	×	✓	×	×
Flash Point	✓	×	×	✓	✓	✓
Kinematic Viscosity	✓	×	×	✓	✓	✓
Carbon Residue	✓	×	×	✓	×	×
Ash Content	✓	×	×	×	×	✓
Lubricity by HFRL	✓	×	×	×	×	×
Copper Strip Corrosion	✓	✓	×	×	×	×
Vapour Pressure	×	✓	✓	×	×	×
Benzene	×	✓	×	×	×	×
Vapour Lock Index	×	✓	×	×	×	×
Appearance	×	✓	×	×	×	×
Oxidation Stability	×	✓	×	✓	×	×
Oxigenates Content	×	✓	×	×	×	×
Ester Content	×	×	×	✓	×	×
Acid Number	×	×	×	✓	✓	×
Iodine Value	×	×	×	✓	×	×
Gliseride Content	×	×	×	✓	×	×
Methanol	×	×	×	✓	×	×
Composition Analysis	×	×	✓	×	×	×
Motor Octane Number	×	×	✓	×	×	×
Temperature at 150 kPa	×	×	✓	×	×	×
Diene Content	×	×	✓	×	×	×
Viscosity Index	×	×	×	×	✓	×
Pour Point	×	×	×	×	✓	✓
Base Number	×	×	×	×	✓	×
Element Analysis (Ca, Mg, Ba, Zn, P)	×	×	×	×	✓	×
Total Residue	×	×	×	×	×	✓

Aim

The scheme is designed for the laboratories that analyses petroleum products in Turkey in order to meet the PT participation requirement of ISO/IEC 17025 standard. PT scheme help laboratories to assess their performances, personnel's competence, methods and/or equipments.

Scope

A twice a year program is being carried out since 2006. There are about 60 participants most of whom are private companies that regularly attend the program.

- **2006 to date:** Gasoline (27 analytes), Gasoil (25 analytes) and LPG (18 analytes)
- **2007 to date:** Biodiesel (18 analytes), Mineral Oil (13 analytes)
- **2011:** Fuel Oil (pilot run)

Sample Preparation

Fuel samples are obtained from the refineries, fuel stations, distributor companies in order to maintain the diversity of the fuel properties. Each laboratory receives two 1 lt capacity bottles of liquid samples and one 2 lt capacity LPG sample. The sampling is performed according to ISO 13528 standard. A random selection is applied for participant samples, homogeneity samples, stability samples and spare samples.

Homogeneity and Stability

ISO 13528 explains the methodology for homogeneity and stability tests. According to the standard, 10 samples are required to be randomly selected among all the samples. Having volatile components in its composition (especially gasoil), the splitting of the samples is inconvenient for fuel samples. Therefore two samples are taken in succession for homogeneity tests.

Program standard deviation values are revised by adding the effect of heterogeneity, if the homogeneity test fails.

Stability of the samples are also tested according to ISO 13528 using randomly selected 3 samples when the participants are expected to do the tests.

Statistical Evaluation

The results from the participants are evaluated using robust analysis explained in ISO 13528. The assigned value, the uncertainty of assigned value are obtained using robust analysis. The program standard deviation used to be determined from the reproducibility values of individual analysis standard methods. Since the participants are able to select their own methods, this method resulted in unfair z-score performance assessments. Therefore, starting with pilot fuel oil cycle, current and past data will be used for the calculation of the program standard deviation via robust analysis.

Confidentiality

Protection of the participants' results and performance is maintained by using a secure unique code for each laboratory. The reports are issued with these codes. Reports and data input are accessed via a password protected web site.

Future Work

In order to improve the scheme and run it at maximum quality standards, PAL is planning to be accredited according to the ISO/IEC 17043 "Conformity Assessment—General Requirements for Proficiency Testing" standard besides trying to answer all the participant laboratories' demands.

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