DEP SPECIFICATION

IMPLEMENTATION OF NEAR-INFRARED SYSTEMS FOR MAIN FUELS BLENDING

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DESIGN AND ENGINEERING PRACTICE

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1. INTRODUCTION

1.1 SCOPE

This DEP specifies requirements and gives recommendations for implementing Near-Infrared (NIR) systems, focusing on:

a) Implementation challenges and key requirements, in particular:

- the special nature of NIR as a model based analytical technique, and
- the complexity of a NIR system, which is composed of analysers and a dedicated organisation to deliver analytical applications;

b) The way a NIR system implementation strategy should be rolled out and the site organisation for accomplishing it, in particular the role of the individuals, the application ownership, the calibrator and the site laboratory;

c) How the NIR analyser shall be designed and installed for off-line, at-line and on-line applications, highlighting the use of standard equipment and the setting of performance targets;

d) Tools that are used to validate the NIR analyser(s), and the frequency and necessity of on-going quality assurance.

This DEP is applicable to:

- Laboratory (off-line or at-line) NIR analysers
- Process (on-line) NIR analysers

NIR is most commonly applied for main fuel blending, in particular gasoline, although its scope of application is much larger. This DEP is dedicated to refinery main fuel blending, where the NIR measurements are applied to the blended fuel sampled after the blend header or from a blended product tank or proportional compositor.

Excluded from the scope of this DEP are NIR calibration procedures and outlier detection – for these, refer to SMS 2965-08 (based on ASTM E1655). This DEP generally follows the validation and QC requirements of SMS 2966-08 (based on ASTM D6122).

This is a revision of the DEP of the same number dated August 2009; see (1.5) regarding the changes.

1.2 DISTRIBUTION, INTENDED USE AND REGULATORY CONSIDERATIONS

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This DEP is intended for use in main fuel blending, e.g., gasoline and diesel, but not fuel oil. This DEP may also be applied in other similar facilities.

When DEPs are applied, a Management of Change (MOC) process should be implemented; this is of particular importance when existing facilities are to be modified.

If national and/or local regulations exist in which some of the requirements could be more stringent than in this DEP, the Contractor shall determine by careful scrutiny which of the requirements are the more stringent and which combination of requirements will be acceptable with regard to the safety, environmental, economic and legal aspects. In all cases, the Contractor shall inform the Principal of any deviation from the requirements of this DEP which is considered to be necessary in order to comply with national and/or local regulations. The Principal may then negotiate with the Authorities concerned, the objective being to obtain agreement to follow this DEP as closely as possible.

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1.3 DEFINITIONS

1.3.1 General definitions

The **Contractor** is the party that carries out all or part of the design, engineering, procurement, construction, commissioning or management of a project or operation of a facility. The Principal may undertake all or part of the duties of the Contractor.

The **Manufacturer/Supplier** is the party that manufactures or supplies equipment and services to perform the duties specified by the Contractor.

The **Principal** is the party that initiates the project and ultimately pays for it. The Principal may also include an agent or consultant authorised to act for, and on behalf of, the Principal.

The word **shall** indicates a requirement.

The word **should** indicates a recommendation.

1.3.2 Specific definitions

Term	Definitions
At-line	The configuration of having the analyser near the process at a plant laboratory, but not connected to the process.
Calibration model or Multivariate calibration model or Model	The mathematical expression or the set of mathematical operations that relates component concentrations or properties to spectra for a set of reference samples – ASTM E1655. A model can be called: • Local, if applied using single site data and spectra only; • Global, if applied using data and spectra from various sites combined.
Inlier	A spectrum residing within a gap in the multivariate calibration space, the result for which is subject to possible interpolation error – ASTM D6122.
NIR analyser or analyser	All piping, hardware, computer, software, instrumentation and calibration model(s) required to automatically perform analysis of a process or product stream – ASTM D6122.
NIR analyser result(s) or analyser result(s)	The numerical estimate of a physical, chemical, or quality parameter produced by applying the calibration model to the spectral data collected by the analyser – ASTM D6122. Also called estimate or predicted primary test method result(s) (PPTMR).
NIR analysis	The process of measuring a spectrum and applying the calibration model to this spectrum, pre-processed as required, so as to estimate a component concentration value or property.
NIR application	A specific analysis by means of a combination of a NIR analyser and a specific process/product sample, manually or automatically, to generate one or more analyser result(s). There can be as many results as reference methods.
NIR calibration or calibration	A process used to create a model relating two types of measured data, a process for creating a model that relates component concentrations or properties to spectra for a set of known reference samples. – ASTM E1655.
NIR Calibrator	The party developing and maintaining the calibration models.

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Term	Definitions
NIR instrument or instrument	Spectrophotometer, associated electronics and computer, spectrophotometer cell and, if utilized, transfer optics – ASTM D6122.
NIR system	Combination of NIR analyser, local staff/organisation, NIR Calibrator and QA/QC procedures covering one or more NIR applications. Also called process analyser system when installed on-line.
Off-line	The configuration of having the analyser at the central laboratory.
On-line	The configuration of having the analyser connected to the process through a sampling system.
Outlier detection methods	Statistical tests which are conducted to determine if the spectrum of the unknown sample falls within the validity range of the model.
Outlier detection results or outlier results	Outcome of the outlier detection method while applying appropriate outlier detection limits or statistics. The analyser results obtained for outliers have unknown accuracy, and might be incorrect. Therefore, the results shall be checked by the PTM.
Outlier sample or outlier	A sample whose spectrum is declared to be an outlier.
Performance test	A test that verifies that the performance of the instrument is consistent with historical data and adequate to produce valid results – ASTM D6122.
Spectrometer cell or cell	An apparatus which allows a liquid hydrocarbon to flow between two optical surfaces which are separated by a fixed distance, i.e, the sample path length, while simultaneously allowing light to pass through the liquid – ASTM D6122.
Validation test or validation	A test performed on a validation sample that demonstrates that the result produced by the analyser and the result produced by the primary test method are equivalent within control limits – ASTM D6122.

1.3.3 Abbreviations

Term	Definition
AMADAS	Analyser Maintenance and Data Acquisition System
APC	Advanced Process Control
BOD	Basis of Design project phase
COA	Certificate of Analysis
DCS	Distributed Control System
DVPE	Dry Vapour Pressure Equivalent
FAT	Factory Acceptance Test
FT-NIR	Fourier Transform Near-InfraRed spectrometer
GFPS	Global Fuel Proficiency Scheme
LIMS	Laboratory Information Management System
NIR	Near-InfraRed (region of light wave number: 4000 - 12500 cm^{-1} equivalent to 780 – 2500 nm, ASTM E131)
OPEX	Operational Expenditure

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Term	Definition
PCA	Principal Component Analysis
PCR	Principal Component Regression
PLS	Partial Least Squares
PQM	Product Quality Management
PT	Proficiency Testing program/scheme
PTM	Primary Test Method
QA	Quality Assurance, procedures applied for system acceptance
QC	Quality Control, procedures applied for system performance monitoring
QE	Quality Estimator
QMI	Quality Measuring Instrument, also known as Process Stream Analyser
R	Reference method precision (Reproducibility) as published in the standard
R'	Site precision according to ASTM D6299.
RMSEC (V)	Root mean square error of calibration (cross validation)
RMSEP	Root mean square error of prediction
RVP	Reid Vapour Pressure
SAT	Site Acceptance Test
SEP	Standard Error of Prediction
SMS	Shell Method Series
SQC	Statistical Quality Control
VRM	Validation Reference Material (ASTM D6122)

1.4 CROSS-REFERENCES

Where cross-references to other parts of this DEP are made, the referenced section number is shown in brackets ( ). Other documents referenced by this DEP are listed in (6).

1.5 SUMMARY OF MAIN CHANGES

This DEP is a revision of the DEP of the same number dated August 2009.

1.6 COMMENTS ON THIS DEP

Comments on this DEP may be sent to the Administrator at standards@shell.com, using the DEP Feedback Form. The DEP Feedback Form can be found on the main page of "DEPs on the Web", available through the Global Technical Standards web portal http://sww.shell.com/standards and on the main page of the DEPs DVD-ROM.

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2. NIR APPLICATIONS AND SYSTEM

2.1 GENERAL

The principles to be recognised when implementing any NIR application are:

1. NIR is a model-based technique. A model is a mathematical expression or set of mathematical operations that relates component concentrations or properties to a pre-selected NIR wavelength domain.

   A NIR analyser predicts a physical, chemical, or quality parameter produced by applying a model to the spectral data (absorbance spectrum) collected by the analyser.

   A NIR analyser may embed one or more calibration models, depending upon the type of application(s).

2. All NIR applications, whether off-line, at-line or on-line, contain a standard instrument and software. A single off-line analyser (per application) should be used to collect spectral data for any on-line application. Analysers and software shall be sourced from Manufacturers that are approved by the Principal.

3. For NIR analysers to deliver reliable analyser results, outlier detection shall be embedded in the NIR analysis. Proper tuning of outlier detection limits shall be performed to flag samples that are outside the validity domain of the model and therefore, have results that are of unknown accuracy. Any sample whose spectrum is declared an outlier shall be analysed by the reference method.

4. NIR is a secondary measurement technique. The implications of this include the following:

   a) The application of NIR is intimately tied to the off-line laboratory measurements by the PTM for model calibration, validation, SQC, and, in some instances, correction. Great attention to these measurements is required for a successful NIR implementation.

   b) Off-line laboratory measurements by the PTM may be reduced in number but cannot be fully eliminated by implementing NIR.

   c) NIR is based on modelling many spectral variables which are tied to variations in fuel compositions and regressed with PTM measurements. This means that to be able to develop accurate models, spanning the blend compositions to be produced, calibrations shall be built using a training set containing many (typically 100-300) samples. Major compositional variations in blended fuels occur not only as a result of variations in the ratios of blending components used, but also from compositional variations within those components. These latter variations are usually not experienced over periods shorter than several months. As a result, it is important that model training set data span all grades, all seasons, (at least) 6-12 months of normal operation of the refinery and, where possible, include periods of conversion unit turnarounds and outages.

   d) Lab-blends should not be used exclusively to prepare training sets since they are generally not representative for real world products.

   e) Errors in NIR results cannot be eliminated simply by making many replicate NIR measurements (averaging) on the same composition, as it will not eliminate model errors. For minor compositional variations over the course of a blend using blending components of nearly constant composition, NIR model errors are nearly constant for some properties.

   f) The use of NIR in making property measurements of blended fuels is, in part, based on the assumption that all of the major compositional changes that affect a given property are reflected in the NIR spectrum of the blended fuel. For some properties, this is not the case (e.g., sulphur) and therefore not all properties can be accurately (or at all) measured by NIR. There are other properties for which the information in the spectrum is poor, and model accuracies may be insufficient to

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meet all measurement needs for those properties.

5. The need for good lab performance.

   NIR model errors can be dominated by PTM measurement precision errors in the training set. The model is generally only as good as the PTM data allows. NIR analyser results can be very precise and accurate and therefore can be used for product certification, provided:

   a) appropriate calibration standards are applied;

   b) appropriate QA/QC procedures are applied (5);

   c) regulations allow its use (3.4);

   d) customers do not prohibit its use.

As a consequence, the Principal shall ensure that:

1. A NIR system (2.3) is set up around any NIR application or group of applications. NIR calibration uses multivariate calibration models requiring a specific organisation (3.6.2) to develop and maintain the applications/models.

2. An implementation strategy (3) is followed to ensure accurate, robust and sustainable NIR applications, in which the proper NIR analyser(s) (4) are designed and set up, and their performance (5) is monitored to ensure they are in-line with their performance targets (3.7.5).

3. The proper NIR application sequence is followed (2.4).

2.2 SCOPE OF NIR APPLICATIONS

Gasoline and diesel blending and the corresponding blending streams are the most common NIR applications.

A NIR application is a specific analysis done by means of a combination of a process sample and a NIR analyser, which can deliver one or many results. There can be as many results as reference methods, but independent for each process.

Any NIR analyser can run many applications, depending on its capability to accommodate different process samples (4). The specific cell design and instrument parameters are specified in (4.2).

2.3 NIR SYSTEM COMPONENTS

For a specific site, the NIR system is a combination of all the NIR analysers, the local staff/organisation, one or more NIR Calibrators, and QA/QC procedures covering all NIR applications.

Unless otherwise specified by the Principal, the composition of such a system shall include the following elements in addition to the analysers:

1. A NIR focal point (3.6.2) who coordinates matters around NIR applications and who is responsible for the NIR performance.

2. A Calibrator, who shall be a person trained to build NIR models.

   There are two alternatives:

   - The site contracts out the calibration to a certified expert (a chemometrician or a by a chemometrician-approved calibrator); or
   - The site performs the calibration, in which case the Calibrator shall:

     a. follow a specified expertise path (3.6.3),

     b. be approved by an authorised expert (chemometrician),

     c. preferably have a back-up available.

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