Good Laboratory Practice in Analytical Laboratory

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Abstract: This article briefly describes the practice in IR-4 analytical laboratories in compliance with EPA Good Laboratory Practice standards. This includes testing facility, protocol and SOP, method development, method validation, sample analysis, storage stability study, linearity, accuracy, precision, limit of detection and limit of quantitation, and archives. [The Journal of American Science 2005;1(2):93-94].

Key Words: Good Laboratory Practice; GLP, IR-4; analytical laboratory; EPA, FDA, OECD; regulatory; registration; linearity; accuracy; precision; LOD, LOQ, SOP; protocol

1. Introduction

Results of scientific research are believed to be objective "truth" through developed knowledge and reliable testing methods. In this respect, regulatory science is no different. Misconduct and carelessness in the research significantly affect human health and the environment [1]. Good Laboratory Practice (GLP) regulations were introduced in 1970s. Before and after 1987, some tolerance petitions were rejected by EPA because of suspected fraudulent data. 40 CFR 160 requires "... good laboratory practices for conducting studies that support or are intended to support applications for research or marketing permits for pesticide products regulated by EPA". This requirement also includes research which is intended to persuade EPA to grant, modify, or leave unmodified a registration or other approval required as a condition of sale or distribution of pesticide.

Nowadays, GLPs are solid standards for government registration and regulatory research facilities. Outside the U.S., OECD GLP is more familiar and has been applied in some other countries [2-3]. It is important that not only proper science must be used in the scientific research but also quality assurance systems should be implemented while we attempt to produce reliable and reproducible data in regulatory research because all of these activities have great impacts on toxicology and the environment.

Interregional Research Project No. 4 (IR-4) is a USDA research program that provides safe and effective pest management solutions for specialty crop growers and submits pesticide safety data on minor crops to EPA for registration. IR-4 has four regional research centers at Cornell University, Michigan State University, University of Florida and University of California Davis, plus USDA ARS research centers. This article described a practical guide to Good Laboratory Practices in IR-4 analytical laboratories.

2. Discussion

GLP projects include usually long-term, pre-determined experiments agreed by sponsors before projects start. GLP research facilities use adequate resources (personnel, space, equipment, and method) to perform GLP research. A Study Director is hired by the management to overview the entire research project while an independent quality assurance unit is setup to perform its quality assurance duties. Testing facility and other components are discussed as follows.

Testing Facility: A GLP research lab is inspected to ensure that the testing facility is GLP compliant prior to GLP research. Key aspects are as followings: chart of organization, personnel, job description, facility layout, protocols, master schedule, Standard Operating procedures (SOPs), records of instrument maintenance, records of standard reference materials, chain of custody records, calibration, method validation, data traceability, corrective actions, archives, etc. Often, a testing facility can apply for OECD GLP recognition to obtain accreditation [2]. Typically, EPA GLP is to inspect the entire testing process.

Protocols and SOPs: Each GLP project has a protocol signed and approved by Study Director and the sponsor and describes how to carry out the study. An amendment is a change in protocol and is planned, expected, and permanent. SOPs describe routine lab activities such as performing extractions and operating instrument. A deviation is a change in GLP or protocol or SOP and is unplanned, unexpected, and temporary.

Reference standard materials: Reference standards are GLP certified. Working standards, including calibration standards, spike standards and internal standards, are prepared in-house by diluting the

reference standards with solvents. Other chemicals meet requirements for testing purposes [4]. Records of preparation of working standard solutions, expiration dates and storage conditions are kept.

Method development: Often the testing facility does not develop a method for the analytical work since a method is always specified in the protocol and may be available. This method may be modified according to the testing facility conditions. Study Director approves modifications to the method.

Method validation (MV): MV is required even if valid methods are used for compliance with analytical work. Fortified samples were prepared by spiking untreated control samples with known amounts of chemicals [4-6]. After going through the sample preparation process, fortified samples are analyzed. Recovery rates are calculated using amount obtained being divided by spiked amount. Typically, the acceptable range of recovery is between 70% and 120%.

Sample analysis: Sample analysis is performed using the above method that has been validated. Typically, an untreated control sample, two treated field trial samples, and a fortified sample (QA sample) are analyzed. Any residue found in the untreated control samples indicates sample contamination or improper testing method. As IR-4 lab practice, residue levels in the untreated control samples should be less than LLMV (lowest level of method validation). QA samples are prepared by spiking untreated control samples with known amounts of the pesticide. This amount of spiking is set to be close to the amount that is expected to be in the treated trial samples. Recovery of QA spikes indicates quality of the testing method under the experimental conditions. Typically, the acceptable range of recovery is between 70% and 120%.

Storage stability study (SS): GLP projects are sometimes long-term research projects. It is known that some pesticides decompose during a certain period of time. Therefore, SS study is to ensure stability of the pesticides during the storage period. SS samples are prepared according to the protocol and lab SOPs. SS samples are stored under the same conditions as treated trial samples. Analyses of SS samples are in the same manner as analyses of the treated samples.

Linearity, accuracy and precision: The linear calibration for analyses is often used [5,7]. The Linearity, accuracy and precision may be used to evaluate the method performance of the analysis that was conducted in the study [6,8].

LOD and LOQ Limit of detection (LOD) and limit of quantitation (LOQ) are important parameters that define the limitations of an analytical method and can also be calculated using statistical methods [6,8].

3. Conclusion

A laboratory that has implemented a quality system has definite advantages when performing testing in compliance with Good Laboratory Practice.

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