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**EVALUATION OF EFFECT OF EXCIPIENTS, TEMPERATURE AND
HUMIDITY ON THE STABILITY OF LEVOTHYROXINE SODIUM**

BY

NIRAJ S. AMIN

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE

REQUIREMENTS FOR THE DEGREE OF

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MASTER OF SCIENCE THESIS

OF

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UNIVERSITY OF RHODE ISLAND

2000

ABSTRACT

Levothyroxine Sodium is one of the most critical drugs for a significant segment of the population. The development of HPLC method of analysis for levothyroxine sodium revealed that there were significant stability problems associated with this drug. On August 14th, 1997, FDA announced that there was evidence which shows significant stability and potency problems associated with orally administered levothyroxine sodium products. This lack of stability and potency has a potential to cause serious health consequence to the public. The literature, on determining the stable formulation of levothyroxine sodium is very little. Much of the published work on levothyroxine sodium describes the innumerable bioavailability and potency problems with this drug. However, little in the literature explored the stability of levothyroxine sodium in presence of various excipients. The portion of work in this thesis represents a series of investigations we have performed to evaluate the stability of levothyroxine sodium in presence of some of the most commonly used excipients at different temperatures and humidities. These studies indicated that levothyroxine sodium is unstable in presence of carbohydrates such as dextrose, lactose and starch. These excipients have an aldehyde group, which may be reacting with the free amino group of levothyroxine sodium leading to a Schiff-base reaction along with oxidation reaction.

In further, series of investigations, the kinetics of degradation reactions were evaluated. First order and bi-phasic first order models were evaluated for our studies. Nonlinear regression was performed using Sigma Plot and the results suggest a biphasic-first order degradation pathway in most of the cases. The shelf life for levothyroxine sodium was

determined using the k-values obtained from the best fit models. The lower t_{90} values indicated that levothyroxine sodium is highly unstable in presence of moisture and higher temperatures and so the tablets should be formulated and stored at or below room temperature with 0% humidity.

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PREFACE

This body of work represents the series of investigations we have performed to determine the stability of levothyroxine sodium in presence of different excipients under various conditions. Section I includes the general introduction regarding the solid state stability of levothyroxine sodium. The main body of this thesis can be found in Section II and Section III. The various chapters in the second and third section can be best envisioned as the series of investigation performed to look at the stability of levothyroxine sodium. Appendix A contains the statistical output of non-linear regression (Sigma Plot, Version 4, SPSS Inc., 1997) in modeling the degradation kinetics of levothyroxine sodium. Appendix B includes the residual plots which were used in checking the adequacy of a fit. At the end of the thesis a bibliography, which sites all the sources used in this document, is included.

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SECTION I: INTRODUCTION

SOLID STATE STABILITY: AN OVERVIEW

Background

Among the different types of drug delivery systems, solid dosage forms are by far the most common due to their dose precision, low cost, easy and inexpensive packaging and shipping. In addition product identification for these dosage forms is simple when done using embossed or monogrammed punch faces; lend themselves to certain special-release profiles, such as enteric or delayed released products; having the best combined properties of chemical, mechanical and microbiological stability of all oral forms and are the lightest and most compact of all oral dosage forms. The advantages of solid dosage forms are reflected by widely accepted practice of delivering drugs in this manner during initial clinical trials [1, 2]. Solid state stability of a drug is one of the most intrinsic and vital parts in the formulation of drug substance. One of the primary concerns during product development is producing a drug substance that has consistent stability properties in the bulk form as well as when formulated [3]. Stability testing provides evidence of how the quality of the drug substance or drug product varies over time under the influence of a variety of environmental factors such as temperature, humidity and light. A systematic approach should be adopted in the testing and evaluation of stability information which should cover, as necessary, physical, chemical, biological, and microbiological quality characteristics, including unique properties of the dosage form (for example, dissolution rate for oral solid dose forms). The design of the stability study is to establish, based on testing a minimum of three batches of the drug product, shelf life and label storage instructions applicable to all future batches of the dosage form manufactured and packed under similar circumstances. The degree of variability of

individual batches establishes the confidence that a future production batch will remain within specification until the expiration date [4]. Stability testing of the drug substances and drug products is required to support the defined expiry period for the following categories of drug regulatory submissions: Investigational New Drug Applications (IND's), New Drug Applications (NDA's) for both the New Molecular Entities (NME's) and non-NME's, Abbreviated New Drug Applications (ANDA's), Supplements and annual reports, Biologics License Application (BLA's) and product license applications (PLA's) [5].

The term 'pharmaceutical stability' encompasses a range of parameters. The most common interpretation is the chemical stability of the drug substance in a dosage form. However, the performance of a drug when given as a tablet, capsule, syrup or injection is not only dependent upon the content of the drug substance, but also on its pharmaceutical properties (dissolution, disintegration, hardness, friability, content uniformity etc.) [1]. All of these parameters must, therefore, be a part of the stability program. In 1994 International Conference on Harmonization (ICH) guidelines for stability testing were published and are as follows [4].

Information on the stability of Active Pharmaceutical Ingredient (API) under defined storage conditions is an integral part of the systematic approach to stability evaluation. Stability information from accelerated and long-term testing should be provided on at least three production batches. Long term stability should cover a minimum of 12 month's duration on at least three production batches at the time of submission. The testing should cover especially those features susceptible to change during storage and likely to influence quality, safety and/or efficacy. Stability information should cover as

necessary the physical, chemical, biological, and microbiological test characteristics. The length of studies and storage conditions should be sufficient to cover storage, shipment, and subsequent use. The 6-month accelerated testing should then be carried out at a temperature at least 15°C above long term storage temperature (25°C ± 2°C, 60% ± 5% RH. The general guidelines for storage conditions and testing periods for bulk drugs and drug product is given in Table 1.

Table 1. Long-term/accelerated testing conditions

	Conditions	Minimum Time Period At Submission
Bulk Drug		
Long Term Testing	25°C ± 2°C, 60% ± 5% RH	12 months
Accelerated Testing	40°C ± 2°C, 75% ± 5% RH	6 months
Intermediate Testing	30°C ± 2°C, 60% ± 5% RH	6 months
Drug Product		
Long Term Testing	25°C ± 2°C, 60% ± 5% RH	12 months
Accelerated Testing	40°C ± 2°C, 75% ± 5% RH	6 months
Intermediate Testing	30°C ± 2°C, 60% ± 5% RH	6 months

If 'significant change' occurs due to accelerated testing, additional testing at an intermediate condition e.g., 30°C ± 2°C, 60% ± 5% RH should be conducted. 'Significant change' at the accelerated condition is defined as [1]:

- A 5 percent potency loss from the initial assay value of the batch;
- Any specific degradant exceeding its specification limit;
- The product exceeding its pH limits;
- Dissolution exceeding the specification limits for 12 capsules or tablets;
- Failure to meet specifications for appearance and physical properties e.g., color, phase separation, resuspendibility, delivery per actuation, caking, hardness etc.

Modes of Drug Degradation

Drug degradation occurs by four main processes [6]:

Hydrolysis due to H_2O , H_3O^+ , OH^- , pH

Oxidation

Photolysis due to UV, visible light

Trace metal ion catalysis due to Fe^{+2} , Fe^{+3} , Cu^{+2} , Co^{+2} , etc.

The decomposition of drugs are most often classified as either hydrolysis or oxidation.

Most drugs contain more than one functional group, and hence may be subjected simultaneously to oxidation as well as hydrolysis. Other reactions such as epimerization, isomerization and photolysis may also affect the stability of drugs.

Hydrolysis may be caused by reaction of water with amides or esters. Water may also react with the ions of salts of weak acids and weak bases. Molecular hydrolysis reactions proceed much more slowly than the ionic hydrolysis (protolysis) [7]. In solid dosage formulations, free moisture is contributed by various additives or excipients, as well as the drug. In tablets a small percentage, typically 2% (w/w) of moisture, is required to facilitate good compression. This free water has the ability to act as a vector for chemical reactions between the drug and the excipients [6]. Some of the most common examples are: the hydrolysis of aspirin above pH 10 and hydrolysis of chloramphenicol which is pH independent is catalyzed by general acids and bases [7].

Oxidation reactions involve the removal of electrons or loss of hydrogen (dehydrogenation) from the molecules. When the reaction involves molecular oxygen, it is called auto-oxidation. Oxidation reactions frequently involve free radicals of atoms or molecules containing one or more unpaired electrons, or free hydroxy and molecular

oxygen ($O - O$). Oxidation may be catalyzed by the presence of trace amounts of heavy metals and organic peroxides [7]. The oxidation of unsaturated fats and oils proceeds in the presence of atmospheric oxygen, light and trace amounts of catalysts according to free radical chain reactions. The hydroperoxide ($R' - CHOOH - CH = CH - R''$) formed in the reaction may further decompose, and the reaction continues until the free radicals formed in the reactions are destroyed by inhibitors or by side reactions which will break the chain. Another classic example is the oxidation of ascorbic acid to dehydroascorbic acid in the presence of copper ions and oxygen. When the solution of ascorbic acid is freed from copper ions, the oxidation of ascorbic acid in alkaline medium will cease [7].

Oxidation and, to some extent, photolysis may be catalyzed by light. The energy of light is inversely related to wavelength (ultraviolet > visible > infrared) and is independent of temperature. When the molecules are exposed to electromagnetic radiation (EMR), they absorb light at characteristic wavelengths, which causes an increase in the energy state of the compound. This energy may cause:

decomposition

retention or transfer of energy

conversion to heat

emission of light at a new wavelength (as fluorescence, phosphorescence).

Photodegradation is dependent on both the intensity and the wavelength of light and is usually mediated by free radicals to produce dark colored substituents [6]. Some of the simple strategies for improving drug stability are given in Table 2.

Table 2. Simple strategies for improving drug stability

(Table reprinted from reference 6)

	Degradation Process	Method of Protection
1)	Hydrolysis	Remove water, moisture e.g. Use dry desiccant
		Lower water activity e.g. Add humectants
		Change excipient(s) e.g. Compatibility studies
		Use solid dosage forms e.g. Tablets and capsules Freeze dry injections
2)	pH	Identify pH _{min} e.g. Addition of buffers
3)	Temperature	Refrigerate 4 C Cool place ≤ 15 C
4)	Oxidation	Include antioxidant e.g. Ascorbic acid Sulphites
		Chelate metal ions e.g. EDTA
		Remove O ₂ e.g. N ₂ , CO ₂ or He
5)	Photolysis	Protect from light, Packaging

Drug-Excipient Compatibility Testing

What emerges from a drug discovery program is an Active Pharmaceutical Ingredient (API) or drug substance. The API is the basis for producing the therapeutic activity expected of the drug and becomes a drug product after formulation with various excipients to produce a dosage form. Hastening the drug development process and optimization of dosage form stability are two major goals of any drug development program. The successful formulation of a stable and effective solid dosage form depends on the careful selection of the excipients used to facilitate administration, promote consistent release, enhance bioavailability of the drug and protect it from degradation. Hence, excipients are the integral components of almost all pharmaceutical dosage forms. In mixtures of solids, incompatibilities or chemical interactions can occur by following mechanisms [6]:

- degradation by nucleation via the gaseous phase

- contracting surface due to nucleation with coverage by the breakdown products

- degradation mediated by surface moisture or eutectic films

- oxidation

- photolysis.

Degradation in the solid state may be affected by several factors, such as the proportion of the drug to excipient(s), method of mixing, hygroscopicity of the powder mixture, hygroscopicity of the substance involved, temperature, humidity, particle size distribution, particle packing, porosity of the powder bed, etc. Hence, in pharmaceutical preformulation drug-excipient compatibility studies are obligatory. Interestingly, with the

importance of drug-excipient compatibility studies, no general method is available for these studies.

Ahlneck et al. [8] studied the three commonly used methods for drug-excipient screening: the suspension technique, storage of powder mixtures and compacts at specified temperatures and stored at specified relative humidities; and evaluated the variables influencing drug degradation. They concluded that the suspension technique is a fast-screening method for detecting chemical stability problems but gives limited information on the stability of a drug in solid dosage forms. The solid state techniques, i.e. the powder mixtures and compacts, gave a better picture of the stability profile of a solid dosage form composition. The solid state procedure took into account a large number of variables such as powder mixing, particle size, surface area, moisture adsorption, etc.

Monkhouse et al. [9] suggested that one should eliminate drug-excipient compatibility testing and instead select excipients on the basis of the physical and chemical characteristics of the drug substance and the literature data for the excipients. They recommended that the final composition should be selected on the basis of the accelerated stability testing of one or more target formulations at high temperature and humidity.

In 1999, Serajuddin et al. [10] reported a method that may be used successfully to identify the relative influence of different excipients on the stability of the drug. They proposed a model which involved storing drug-excipient blends with 20% added water in a closed glass vials at 50°C and analyzing at one and three weeks for chemical and physical stability. The amount of the drug substance in the blend was determined on the basis of the expected drug-to-excipient ratio in the final formulation. The effect of several

key factors such as the chemical nature of the excipient, drug-to-excipient ratio, moisture, micro environmental pH of the drug-excipient mixture, temperature, and light on the dosage form stability could be identified by using this model. They suggested that selection of the dosage form composition by using this model at the outset of the drug development program would lead to a reduction of surprise problems during long term stability testing of drug products.

Quality and Functionality of The Excipients

Excipients are better known as promoters of degradation rather than as stabilizers of drug substances. Different functional groups or residues present in the excipients may have the propensity to interact with labile active ingredients or drugs, causing loss of molecular integrity or degradation. The quality of the final product depends not only on the active principles and production processes, but also on the performance of the excipients. Excipients have undergone an evolution from an 'inert' and cheap vehicle to an essential constituent of the formulation that enhances the stability and the bioavailability of the drug substance in the drug product and improves its manufacturability on a production scale. The studies of interactions between a drug and the excipients shows that complexation, hydrogen bonding, ion-dipole, dipole-dipole and van der waals attractions can modify the physicochemical, pharmacological or pharmacokinetical behavior of the final product especially in the solid dosage forms [11, 12, 13].

If excipients have to act as stabilizers they must obviate or alternate the factors that cause molecular transformation of drug substances. These factors may be environmental components such as water vapor and sunlight. Other factors include stress during the

processing of the dosage form such as size reduction, compaction or sterilization, or interactions between adjacent molecules of the drug or functional groups on the same molecule [11].

Moisture Related Degradation

Water is one of the major factors responsible for causing degradation in pharmaceutical formulations. It may be associated with the drug or the excipients, may be incorporated during the processing of dosage forms or may be acquired from the environment during packaging or storage. Because of its ubiquitous nature and its ability to exist as a vapour, water is virtually impossible to avoid and difficult to control. The molecular mass of water is low, and so small amounts may be significant in terms of molecular reactivity. Water is also capable of diffusing, to some extent, through packaging materials, pack seals or through compacted solid dosage forms [11].

Excipients with a greater affinity for moisture might aid in mitigating moisture sensitivity. Thus, formulation with a substance having a greater affinity for water as compared to the drug may help in sequestering moisture in the product. Perrier et al. [14] used nitrogen sorption isotherms to predict the effect of common excipients on the stability of nitrazepam. They determined that excipients with higher absorption energies caused less degradation, meaning, if the excipient has a higher binding energy for water as compared to the drug, the excipient may act as a desiccant and stabilize the drug. Along with moisture, residues of lower alcohols (methanol, ethanol, isopropanol) might be present in the final formulation, as a result of synthesis, isolation of the drug or the process used for manufacturing the dosage form. Nimry et al. [15] and Tobyn et al. [16]

showed that materials such as amorphous silica and microcrystalline cellulose may act as 'scavengers' of volatile residues and help in stabilizing the formulation.

Degradation by Oxidation

Loss of drug quality due to oxidation is usually secondary to hydrolytic breakdown. These reactions are often complex and caused by factors that are difficult to separate and clarify. Oxidation can be catalyzed by exposure to air or light, the presence of trace residues (metal ions), by other components in the formulation or the combination of all the above mentioned factors.

Formulation additives have been effective in the stabilization of vitamin preparations. The antioxidants tocopherol, butylated hydroxy anisole, butylated hydroxy toluene and propyl gallate have all been used to stabilize vitamins A and D₃ [17, 18]. Reyes [19] showed that magnesium, calcium and aluminium stearates helped in the stabilization of ascorbic acid.

Photodegradation

Exposure to light may precipitate a plethora of degradation reactions, such as polymerization, isomerization, addition reactions in unsaturated systems, substitution reactions and photo-oxidation [20].

Thoma and Klimek introduced the concept of spectral overlay. This approach involves formulating with an excipient whose UV absorption spectrum overlaps (or substantially overlaps) that of the compound requiring stabilization. The excipient would thus compete with the active compound for photons from a radiation source and hence the impact of damaging radiation would be attenuated. They showed that the photolabile calcium antagonist nifedipine can be stabilized by the natural food colorant, curcumin, or by riboflavin [21]. Sanderson et al. [22] showed that the stability of a β -lactam BRL42715B

can also be enhanced by addition of a 'blocker' such as titanium dioxide and addition of soft paraffin with a UV spectrum that provided a partial spectral cover.

The spectral overlay approach is an elegant way to stabilize a drug. However the list of potentially useful materials which are free from pharmaceutical activity and are non-toxic is very limited.

Other Modes of Degradation

Some degradation reactions like isomerization, dimerization, polymerization and other forms of molecular rearrangements do not involve species other than the active ingredient. These reactions are common in drugs of large molecular mass or those of biological origin. Hence it might seem that molecules with an intrinsic 'self-destructing' capability would be most difficult to stabilize.

Cyclodextrins are unique compounds, due to their unique, molecular complexation capability has been shown to improve the stability of compounds such as clofibrate and isosorbide which have tendency to sublime [23]. Cyclodextrins have also been shown to stabilize labile materials such as PGE₁ and PGF₂ by forming molecular encapsulation [24].

The use of excipients to stabilize an unstable ingredient is an attractive concept. A product can be developed that will retain its quality while the drug and other formulation ingredients are in close association. However, there are few examples, as listed in Table 3, in which excipients may destabilize or decrease the efficacy of the drug.

Table 3. Studies of interactions between API's and excipients in pharmaceutical formulation

Active Medicament/ Excipient	Stoichiometry (molar ratios percentages)	Therapeutic activity	Pharmaceutical formulation	Results of interaction	References
Acetylsalicylic acid/Eudragit RS, starch, Dextrose monohydrate	Different proportions – 5, 10, 15, 25%	Analgesic antipyretic	Matrix-tablets	Poor drug release	Ref. 25
Carteolol hydrochloride/ Eudragit L	22% Carteolol	A potent β -adrenergic blocking action	Tablet	Type ammonium salt interactions (Polymeric complex)	Ref. 26
Chloramphenicol stearate/ colloidal silica		Antibiotic	Powder	In vitro higher enzymatic hydrolysis rate	Ref. 27

Methods for Studying Interactions between Drug and Excipients in Pharmaceutical Formulations

Presently, infra-red (IR)-spectroscopy, nuclear magnetic resonance (NMR) spectroscopy, differential scanning calorimetry (DSC) and X-ray diffraction are the most commonly used methods to investigate interactions between drugs and the excipients in pharmaceutical formulations. IR spectroscopy is sensitive to crystalline form changes. Since it is inherently based on molecular vibrations, IR has an advantage of being sensitive to functional group changes in low or non-crystalline materials. IR spectroscopy helps in looking at interactions between the drug and the excipient by following important characteristics such as the appearance of new IR absorption band(s), the broadening of band(s), and alteration in intensity. These comparisons are performed by comparing the IR spectra of the drug alone, the excipient alone, the complex and the simple physical mixture (prepared in same stoichiometry as a complex from both the drug and the excipient(s) [13]. For example, IR was used to determine which excipients participate in the salt to free base conversion of delavirdine. The spectra of delavirdine mesylate indicated that free base was formed in all the mixtures except the one where croscarmellose sodium was absent. The spectra revealed that croscarmellose sodium in the tablet matrix was necessary to prevent conversion of the delavirdine salt to a free base [27]. An electronic database is available for comparison and identification of absorption and transmission spectra for excipients and the drugs which can be used to determine functional groups or degradation products present in the mixtures. This can be done by extrapolating the spectra obtained with that present in the database.

Solid state NMR can be used to determine inter and intra-molecular interactions and crystal packing in the solids at a molecular level; and are, therefore, useful for distinguishing between closely related solid forms of the same molecular entity. Rohrs et al. [28] used solid state NMR to characterize and quantify the forms of delavirdine in the tablet form. Three crystalline forms of delavirdine mesylate have been characterized, and the distinct NMR features observed among these and other forms were used for diagnostic purposes. The identification of the drug form(s) were used to verify that no major interferences occurred from tablet excipients. It was seen that there were no interferences from the tablet excipients with the drug forms in the tablet matrix.

DSC is another important tool which is used to study changes in the melting point of compounds and hence determine polymorphic changes in the mixtures. Jain et al. [29] used DSC to evaluate the amount of moisture pickup in a drug-moisture-lactose interaction. They found that stable solid lactose exists in α - monhydrous, α - anhydrous, and β -anhydrous forms. DSC studies revealed that the α - anhydrous lactose which is present in small quantities in β -lactose is responsible for moisture uptake and hence makes the formulation unstable.

Crystalline materials in powder form exhibit highly characteristic X-ray diffraction patterns in which the positions and relative intensities of peaks are well-defined and reproducible. The diffraction pattern of a crystalline powder is characteristic for the crystal lattices of that particular polymorph. By measuring the rate of disappearance (or appearance) of a peak unique to a reactant (or product), the kinetics of a reaction or transformation can be determined. This method has been used to follow the desolvation reaction of several crystalline hydrates. Since crystals serve as unique micro-reaction

vessels, X-ray crystallography can often be used to determine the exact position of atoms as they re-orient themselves during a reaction. This can provide valuable information in predicting the path followed by atoms throughout the course of a reaction. Because of the wealth of information offered by X-ray analysis, it can be expected to play an important role in solid state stability studies [30].

LEVOTHYROXINE

INTRODUCTION

In 1891, G.R.Murray published a paper in the British Medical Journal showing the beneficial effects of using sheep thyroid extract in the treatment of hypothyroidism [31]. Kendall [32] and Harrington [33] later determined that levothyroxine was the primary active component in thyroid extracts.

Levothyroxine Sodium is a sodium salt of the levo isomer of the endogenous secretion of the thyroid gland thyroxine. Thyroid hormones affect protein, lipid, and carbohydrate metabolism; growth; and development. Thyroid hormones stimulate oxygen consumption by most of the cells in the body, resulting in an increased energy expenditure and heat production. It also possesses a cardiostimulatory effect that may result in direct stimulation of the heart [34]. Orally administered levothyroxine sodium is used as a supplemental or replacement therapy in conditions with diminished or absent thyroid function such as cretinism, myxedema, nontoxic goiter or hypothyroidism. It is also used as replacement or supplemental therapy in patients with secondary (pituitary) or tertiary (hypothalamic) hypothyroidism, making levothyroxine a life long medication [35]. In

1997, it was estimated that about 8 million Americans received thyroid replacement therapy, which makes it a drug of critical importance for a significant segment of population [36].

In 1955, Flint laboratories introduced the first orally administered synthetic levothyroxine sodium in the United States [37]. In 1982, the FDA, in conjunction with the United States Pharmacopeial Convention, took an initiative in organizing a workshop to set the standard for the use of High Pressure Liquid Chromatography (HPLC) assay for the quality control of levothyroxine sodium. The assay method initially used in the USP was based on a titrametric determination of iodine content, was deemed to be neither specific nor stability indicating and was unable to distinguish between active drug and the iodine containing decomposition products. Moreover, the iodine content method was quite insensitive, requiring 3 mg of levothyroxine sodium per assay or 120 tablets of the 25 ug dosage form which made the method unsuitable for product quality control, regulatory control, content uniformity and routine stability testing [38].

In the quest to find a stability indicating method, Smith et al. [39]. In 1981, proposed the first HPLC system for the determination of levothyroxine sodium in tablets. They used an octadecylsilane reversed phase column, with a mobile phase consisting of potassium dihydrogen phosphate, methanol, and water at 44°C and using a UV spectrophotometric response at 254 nm. Though this method exhibited accuracy and precision, it was not stability indicating, and the quantitation of levothyroxine sodium was complicated by interferences observed in aged samples. In 1984, Garnick et al. [38] proposed a HPLC system which consisted of a bonded phase cyanopropyl column with a mobile phase consisting of acetonitrile : water (40 : 60 v/v) and 0.05% phosphoric acid; and used a UV

detector at 225 nm. After several modifications to this method the USP published a stability indicating assay for levothyroxine sodium and determined that orally administered levothyroxine sodium products had significant stability problems [40].

In 1990, Gupta et al. [41] analyzed levothyroxine sodium tablets from two different manufacturers. The results indicated that one of the brands, contained excipient(s) which acted as catalyst(s) to hasten decomposition. Tablets from the same manufacturer but from different batches showed an additional long peak in the chromatogram, indicating that the excipient(s) may have been changed. In 1992, Won [42], published a most interesting paper, which indicated that the kinetics of degradation of levothyroxine in aqueous solution and in the solid state are significantly different. It appears that in aqueous solution the major degradation pathway involves deiodination while in solid state levothyroxine follows a deamidation pathway.

Between 1987 and 1994, FDA received 58 adverse drug experience reports associated with the potency of orally administered levothyroxine sodium products. Out of the 58 reports, 47 suggested that the products were sub-potent, while 9 suggested super-potency. Some of the problems reported were the result of switching products from different manufacturers. However, other adverse events occurred when a patient received a prescription refill of a product from the same manufacturer on which they had previously been stabilized, indicating a lack of consistency, potency and bioavailability between different lots of tablets from the same manufacturer. From 1991 to 1997, there had been no less than 10 firms initiating recalls of levothyroxine sodium tablets, involving 150 lots and more than 100 million tablets. In all but one case, the recalls were initiated because the tablets were found to be either sub-potent or potency could not be assured through the

expiration date. The remaining recalls were initiated on the products that were found to be superpotent. It appears that the stability problem for levothyroxine sodium is quite complex and with some products, at least, stability is batch dependent [35].

After evaluation of the above mentioned problems, on August 14th, 1997, the FDA stated that there was sufficient evidence which showed significant stability and potency problems associated with levothyroxine sodium products. Usage of such products is believed to cause potentially serious health consequences in the public. In view of the stability and potency problems associated with levothyroxine sodium products, FDA retrograded all levothyroxine products as new drugs for which approval by NDA or ANDA would be required [35].

From the literature, it can be seen that very little has been done to successfully develop a stable formulation of levothyroxine. There are limited publications exploring the stability of levothyroxine sodium in presence of excipients. Investigations to evaluate the stability of levothyroxine sodium in presence of some of the most commonly used excipients, such as dextrose, dicalcium phosphate dihydrate, calcium sulfate, mannitol, etc at various temperatures and humidities would assist in selecting formulation ingredients, and would also provide insight into the nature of degradation typical with levothyroxine sodium. Such studies were hoped to provide a better understanding of the formulation parameters to be used to optimize formulations and processing for the improvement of the drug stability.

GENERAL PROPERTIES OF LEVOTHYROXINE (Ref. 43)

i) *Chemical Name:*

- a) Sodium derivative of 3-[4-(4-hydroxy-3,5-diiodophenoxy)-3,5-diiodophenyl]-L-alanine.
- b) L-3,3',5,5'-Tetraiodothyronine, sodium salt, pentahydrate.
- c) β -[(3,5-diiodo-4-hydroxyphenoxy)-3,5-diiodophenyl]-alanine, sodium salt pentahydrate.

ii) *Generic Name:* Sodium Levothyroxine

iii) *Trade Names:* Synthroid®, Levoxyl®, Eltroxin® etc.

iv) *Empirical Formula and Molecular Weight:*

L-4 Sodium salt pentahydrate

$C_{15}H_{10}I_4NNaO_4 \cdot 5H_2O$ 888.96

Anhydrous Sodium salt of L-4

$C_{15}H_{10}I_4NNaO_4$ 798.86

L-4 free acid

$C_{15}H_{11}I_4NO_4$ 776.93

v) *Structure:*

Figure 1. Structure of levothyroxine sodium

vi) *Organoleptic Properties:*

Color: White to Pale Yellow.

Odor: Odorless.

vii) *Melting Range:*

235°C – 236°C.

viii) *Stability:*

Levothyroxine is sensitive to irradiation, hydrolysis, oxidation and heat.

ix) *Solubility:*

Table 4. Solubility profile of levothyroxine sodium in presence of different solvents

Solvent	Levothyroxine Solubility in gm/ 100ml
H ₂ O	0.14
95% Ethanol	0.4
Alkali Hydroxides	Soluble
Chloroform	Almost insoluble
Ethyl Ether	Almost insoluble
Phosphate buffer pH 7.4	0.022 - 0.044

SECTION II : EXPERIMENTAL

The study of drug-decomposition kinetics, the development of a stable dosage forms, and the establishment of expiration dates for drug products should evaluate the parameters that are likely to affect the quality, safety, and/or efficacy of the drug product. The analytical procedure should be fully validated and the assays should be stability-indicating.

During pre-formulation stage, one of the objectives of the stability studies is to establish excipient-compatibility information to help the formulator design a stable and efficacious formulation. Excipients can affect the stability of drugs by: (1) acting as surface catalysts; (2) altering the pH of the moisture layer; and (3) undergoing direct chemical reaction with the drug. The above mentioned problems thus require a screening of a large number of excipients under several storage conditions such as temperature, humidity, and light. This study helps in developing analytical, physical, and chemical data that will facilitate formulation studies and manufacturing. These studies also allows the formulator to: (1) determine reactivities of the drug substances; (2) establish whether or not special handling and storage procedures are required to protect the drug substance; (3) ensure that the potency is sufficient and the level of significant degradation products may be significant throughout the life of the supplies; (4) develop supporting data for subsequent stability studies of the formulated drug; and (5) delineate any interaction between selected formulation excipients and the drug substance.

Once the initial screening of the excipients is done, the next step is to perform the stability studies on the formulated dosage form(s) under several storage conditions of temperature and humidity. In case of tablets, stability studies should also include

evaluation of characteristics such as: appearance, friability, strength, hardness, color, disintegration and dissolution.

D) STABILITY INDICATING ASSAY FOR LEVOTHYROXINE SODIUM:

The selected HPLC method was selected to provide high sensitivity for levothyroxine sodium along with its reference standard liothyronine sodium at very low concentrations, as well as to be highly reliable and simple to use within the constraint of these studies.

REAGENTS / EQUIPMENTS USED:

Equipments

Metler AE 240 Weighing balance

Ultrasonifier (Fisher Scientific)

Magnetic Stirrer (Thermix® Model 120 MR)

HPLC System consisting of:

Automated Gradient Controller (Waters®)

HPLC Pumps (Waters® Model 515)

Auto Sampler (Waters® Model 717 plus)

LC Spectrophotometer (Waters® Model 480)

Data Module (Waters® Model 746)

L-10 column (Zorbax®)

Column Temperature Controller (Fiatron Systems Inc, Model TC-50)

Reagents

Levothyroxine Sodium (Biochemie Labs, Lot# 78459003)

Liothyronine Sodium (Sigma Labs, Lot # 98H11681)

Sodium Hydroxide (Fisher Scientific, Lot # S318-500)

Methanol (HPLC Grade, Fisher Scientific, Lot # A452-4)

Acetonitrile (HPLC Grade, Fisher Scientific, Lot # A998-4)

O-Phosphoric Acid (Fisher Scientific, Lot # A260-500)

Deionized water.

STANDARD PROCEDURE:

If the HPLC system has not been used for a week or more, the system should be purged and primed as described in the instruction manual.

PREPARATION OF MOBILE PHASE:

Prepare a mixture of water and acetonitrile (70 : 30) containing 1 ml of o-phosphoric acid in each 1000 ml of the mixture. While making the mobile phase water and o-phosphoric acid should be mixed prior to the addition of acetonitrile. Mix the mobile phase for 5 minutes with magnetic stirrer. Filter the mobile phase using vacuum pump. Degas the mobile phase in ultra-sonifier for 5 minutes.

PREPARATION OF HPLC SYSTEM:

Connect the L-10 column to the system and make sure that the connections are tight.

Turn on the system and set the following parameters:

The column thermostat temperature should be set to 30°C.

The absorbance of the spectrophotometer should be set to 225 nm.

The flow rate should be set to 1 ml/minute.

The run time should be set to 20 minutes.

Injection volume should be set to 25 µl.

Number of injections from each vial should be set to 5.

Allow the system to stabilize for at least 2 hours.

PREPARATION OF STANDARDS:

0.01M METHANOLIC NaOH: Approximately 400mg of NaOH was weighed and dissolved in 500ml of water in 1000ml volumetric flask. To it 500ml of methanol was added to make up the volume.

STOCK SOLUTION (500 μ g/ml) OF L-4: Approximately 0.025g of L-4 was weighed and transferred to 50 ml volumetric flask. The volume was made up with 0.01M methanolic NaOH.

STANDARD SOLUTION (5 μ g/ml) OF L-4: 1ml of above stock solution was taken in 100ml volumetric flask and the volume was made up with 0.01M methanolic NaOH.

L-4 SOLUTION (10 μ g/ml): 1ml of above stock solution was taken in 50 ml volumetric flask and the volume was made up with 0.01M methanolic NaOH.

Following the above procedure, L-3 solutions were prepared.

SERIAL DILUTIONS:

L-4 (0.2 μ g/ml) and L-3 (0.2 μ g/ml): 2ml of L-4 (10 μ g/ml) + 2ml of L-3 (10 μ g/ml) in 100ml volumetric flask and volume made up with 0.01M methanolic NaOH.

L-4 (0.4 μ g/ml) and L-3 (0.4 μ g/ml): 4ml of L-4 (10 μ g/ml) + 4ml of L-3 (10 μ g/ml) in 100ml volumetric flask and volume made up with 0.01M methanolic NaOH.

L-4 (0.6 μ g/ml) and L-3 (0.6 μ g/ml): 60 μ L of L-4 stock + 60 μ L of L-3 stock in 50ml volumetric flask and volume made up with 0.01M methanolic NaOH.

L-4 (.8 µg/ml) and L-3 (.8 µg/ml): 80 µL of L-4 stock + 80 µL of L-3 stock in 50ml volumetric flask and volume made up with 0.01M methanolic NaOH.

L-4 (1 µg/ml) and L-3 (1 µg/ml): 100 µL of L-4 stock + 100 µL of L-3 stock in 50ml volumetric flask and volume made up with 0.01M methanolic NaOH.

L-4 (2 µg/ml) and L-3 (2 µg/ml): 200 µL of L-4 stock + 200 µL of L-3 stock in 50ml volumetric flask and volume made up with 0.01M methanolic NaOH.

L-4 (4 µg/ml) and L-3 (4 µg/ml): 400 µL of L-4 stock + 400 µL of L-3 stock in 50ml volumetric flask and volume made up with 0.01M methanolic NaOH.

L-4 (6 µg/ml) and L-3 (6 µg/ml): 600 µL of L-4 stock + 600 µL of L-3 stock in 50ml volumetric flask and volume made up with 0.01M methanolic NaOH.

L-4 (8 µg/ml) and L-3 (8 µg/ml): 800 µL of L-4 stock + 800 µL of L-3 stock in 50ml volumetric flask and volume made up with 0.01M methanolic NaOH.

L-4 (10 µg/ml) and L-3 (10 µg/ml): 1000 µL of L-4 stock + 1000 µL of L-3 stock in 50ml volumetric flask and volume made up with 0.01M methanolic NaOH.

RECOMMENDATIONS:

Confirming to the standard procedure described above, together with the following recommendations allowed the assay of levothyroxine sodium in a consistent and reproducible manner.

Prepare fresh standards everyday.

Wait for the HPLC system to stabilize.

The mobile phase should be properly degassed and the pump should be free of air bubbles. If there are erratic changes in the pump pressures, it might be due to entrapment

of air in the pump heads. The pumps should be primed to remove the entrapped air. If the problem still persists, inlet and outlet check valves of the pump should be checked.

Always mount the magnetic stirrer in the mobile phase.

Any problem with the assay may be due to pumps, column, standard, sample, technique and should be isolated by checking each in turn.

A) VALIDATION OF ASSAY:

Investigations into the validity of the method have been undertaken. Using suitable standards, all containing liothyronine, the slope, measuring range and linearity of the calibration curve were determined for levothyroxine sodium. Additional studies were also completed to determine the validity of the typical analytical parameters used in the assay such as precision, accuracy, linearity, selectivity etc. In a further series of investigations, the storage of levothyroxine sodium in .01 M methanolic NaOH at room temperature over a period of 2 days was also investigated. This was performed to make sure that levothyroxine sodium does not degrade during the analysis, which takes 35 hours.

PRECISION: The precision of an analytical method is the closeness of the test results when the procedure is applied repeatedly to multiple aliquots from a single homogenous sample. Thus, it is a measure of the degree of reproducibility of the analytical method under normal operating circumstances. Precision is determined by repeatedly assaying multiple samples removed from the homogenous sample.

ACCURACY: The accuracy of an analytical method is the closeness of test results obtained by the method to the true value. It is a measure of the exactness of the analytical

method. Accuracy is determined by using the calibration curve to determine the concentration of a sample (with known concentration). The accuracy of our assay method, expressed as percent recovery was found to be greater than 95% throughout the linear range used.

LINEARITY: The linearity of an analytical method is its ability to elicit test results that are directly, or by a well defined mathematical transformation, proportional to the concentration of analyte in samples within a given range. Linearity expressed in terms of the variance around the slope of the regression line.

ACCURACY SELECTIVITY / INTERFERENCES: Placebo analysis was performed to check for interference or junk peaks due to excipients. No interfering peaks were observed with all nine excipients.

REPRODUCIBILITY: Reproducibility is limited by factors such as temperature, fluctuations and noise. The reproducibility was found to be independent of concentration and temperature, as the temperature of the column was controlled by a thermostat.

LIMIT OF DETECTION (LOD): Determination of the signal-to-noise ratio is performed by comparing measured signals from samples with known low concentrations of analyte with those of blank samples and establishing the minimum concentration at which the analyte can be readily detected. A signal-to-noise ratio between 3 or 2:1 is generally considered acceptable for estimating the detection limit. In our assay method LOD was found to be 0.4µg/ml, with a signal-to-noise ratio of 3:1.

LIMIT OF QUANTITATION (LOQ): Determination of the signal-to-noise ratio is performed by comparing measured signals from samples with known low concentrations of analyte with those of blank samples and establishing the minimum concentration at

which the analyte can be readily quantified. A typical signal-to-noise ratio is 10:1. In our assay method LOQ came out to be 0.6µg/ml, with a signal-to-noise ratio of 10:1.

No significant differences were found in the concentrations of the samples stored for 2 days at room temperature. Placebo analysis were performed for all excipients to be used to make sure that the excipient peaks do not interfere with the levothyroxine peaks. No additional peak were seen which confirmed that the excipients did not interfere with levothyroxine peaks. Tables 5 and 6 shows the mean values of the data generated out of five days of validation studies for ten separate and distinct samples for series of levothyroxine sodium and liothyronine sodium concentrations ranging from 0.2 to 10 µg/ml. Figures 2 and 3 illustrate the complete as well as linear portion of the calibration curve for these samples. The correlation coefficient was found to be 0.9999 and 0.9997 for levothyroxine sodium and liothyronine sodium respectively.

Table 5. Reproducibility of levothyroxine sodium from standard solutions over 5 days of validation studies

DAY 1,2,3,4&5	AVERAGE	Std Dev	%CV
0.2	23155.8	1692.483	7.30911
0.4	51158.36	2037.795	3.983307
0.6	84070.32	3589.236	4.269326
0.8	112283.96	5918.373	5.270898
1	139962.48	4930.562	3.522774
2	293628.6	6953.736	2.368208
4	600607.8	12771.75	2.126471
6	904545.04	27886.95	3.082981
8	1215639.2	30161.43	2.481117
10	1496429.92	43786.46	2.926062

Figure 2. Representative calibration curve from the validation study for levothyroxine sodium assay

Table 6. Reproducibility of liothyronine sodium from standard solutions over 5 days of validation studies

DAY 1,2,3,4&5	AVERAGE	Std Dev	%CV
0.2	22956.28	1132.168	4.931844
0.4	52606.12	3601.949	6.847016
0.6	64867.24	7805.121	12.03245
0.8	102057.24	7582.398	7.429554
1	137360.4	7228.503	5.262436
2	302628.04	4502.396	1.487766
4	622099.6	4146.621	0.666553
6	937045.56	9922.515	1.058915
8	1241169.04	17554.86	1.414381
10	1573166.8	13105.93	0.833092

Figure 3. Representative calibration curve from the validation study for liothyronine sodium assay

B) NORMALIZATION OF LEVOTHYROXINE SODIUM

Levothyroxine sodium obtained from Biochemie, Inc. was compared for purity with the standard levothyroxine sodium obtained from Sigma Chemical Company (Lot # 088H1318) which had a purity of 99.1% using our validated HPLC method.

Three samples each containing 5µg/ml of levothyroxine sodium obtained from Sigma Chemical Company and Biochemie, Inc. were prepared using the standard method. The samples were analyzed by HPLC and the results compared using a two sided t-test with 95% confidence interval as shown in Table 7 and Table 8. It was seen that there was no significant difference between the levothyroxine sodium obtained from Sigma Chemical Company and that obtained from Biochemie, Inc.

Table 7. Normalization data for levothyroxine sodium obtained from Sigma chemical company (Standard) and Biochemie, Inc. (Sample)

Standard#1 (5ug/ml)		Standard#2 (5ug/ml)		Standard#3 (5ug/ml)	
AUC	RetentionTime	AUC	RetentionTime	AUC	RetentionTime
708356	15.16	727869	14.96	744958	14.93
725036	15.12	742935	14.96	747136	14.91
734825	15.06	749291	14.94	739102	14.94
737402	15.04	746485	14.94	745375	14.96
AUC	MEAN	AUC	MEAN	AUC	MEAN
RT	726404.75	RT	741645	RT	744142.75
	15.095		14.95		14.935

Sample#1 (5ug/ml)		Sample#2 (5ug/ml)		Sample#3 (5ug/ml)	
AUC	RetentionTime	AUC	RetentionTime	AUC	RetentionTime
736790	15.02	745838	14.94	716068	14.96
750280	15.01	754013	14.94	741785	14.97
742310	15	768462	14.94	748421	14.96
759268	14.96	746411	14.92	754361	14.96
AUC	MEAN	AUC	MEAN	AUC	MEAN
RT	747162	RT	753681	RT	740158.75
	14.9975		14.935		14.9625

Table 8. t-Test: two-sample assuming equal variances for Sigma and Biochemie.

	<i>Sigma</i>	<i>Biochemie</i>
Mean	737397.50	747000.58
Variance	92190103.00	45732353.00
Observations	3.00	3.00
Pooled Variance	68961228.00	
Hypothesized Mean Difference	0.00	
df	4.00	
t Stat	-1.42	
P(T<=t) one-tail	0.11	
t Critical one-tail	2.13	
P(T<=t) two-tail	0.23	
t Critical two-tail	2.78	

II) STABILITY STUDIES FOR LEVOTHYROXINE SODIUM

Evaluation of a stable solid dosage formulation often begins with drug-excipient compatibility studies. The present study was designed to investigate the stability of levothyroxine as a pure drug as well as in the presence of different excipients at different temperatures and humidities. From results of these investigations, excipients as well as optimal formulation and processing conditions were identified that would yield a more stable and reliable dosage form of levothyroxine. Of the many excipients available in the market, selection for this study was based on the physical and chemical properties. Nine excipients were selected for inclusion in the study. The studies were divided into four sections:

- 1) Use of saturated salt solutions to achieve specified relative humidities.
- 2) Stability testing of 900 µg of levothyroxine sodium (pure drug).
- 3) Drug-Excipient compatibility studies.
- 4) Stability studies for levothyroxine sodium tablets.

Samples were placed under different conditions in dessicators to maintain different conditions such as $25^{\circ}\text{C} \pm 2^{\circ}\text{C}$, $25^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and $60\% \pm 5\%\text{RH}$, $40^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and $75\% \pm 5\%\text{RH}$, 50°C with 20% w/w moisture and 50°C (without addition of moisture) and samples were removed at various intervals and analyzed.

METHODS AND MATERIALS:

MATERIALS:

Plastic dessicators (Fisher Scientific), HPLC System(Waters, Inc.), Magnetic Stirrer (Thermix® Model 120 MR), Weighing balance (Metler, Model # AE 240), Temperature and Humidity Logger (Dickson, Model # TL120), Sodium Chloride (AR Grade, Fisher Scientific, Lot # S640-3), Sodium Bromide (AR Grade, Sigma Labs, Lot # S45-47), Dextrose (AR Grade, Fisher Scientific, Lot # S640-3), Sodium Hydroxide (Fisher Scientific, Lot # S318-500), Methanol (HPLC Grade, Fisher Scientific, Lot # A452-4), Acetonitrile (HPLC Grade, Fisher Scientific, Lot # A998-4), O-Phosphoric Acid (Fisher Scientific, Lot # A260-500), Lactose Monohydrate (Quest International, Lot # MRP867682), Lactose Anhydrous (Quest International, Lot # M018151), Ferric Oxide (Fluka Chemicals, Lot # 44955), Mannitol (SPI Polyols, Inc. Lot # 3127G9), Starch 1500 (National Starch and Chemical Company, Lot # CB7137), Calcium Sulfate (Mendell, Lot # 8072CX), Di Calcium Sulfate Dihydrate (Mendell, Lot # X26AX), Talc (Cyprus Industrial Minerals Co., ID # ACM-189-7-03) and Deionized water.

PREPARATION OF SATURATED SALT SOLUTIONS FOR MAINTAINING SPECIFIED RELATIVE HUMIDITIES:

Saturated salt solutions are used to maintain specified relative humidities in closed chambers. Different salts are used to prepare saturated salt solutions, which can be used to obtain a wide range of relative humidities within the different temperature intervals suitable for stability studies of pharmaceuticals [44].

Saturated salt solutions of NaBr and NaCl were prepared to provide the relative humidity levels of $60\% \pm 5\%RH$ and $75\% \pm 5\%RH$ in storage dessicators at $25^{\circ}C \pm 2^{\circ}C$ and $40^{\circ}C \pm 2^{\circ}C$ respectively.

Experimental Design: To obtain the humidity of $60\% \pm 5\%RH$ at $25^{\circ}C \pm 2^{\circ}C$ and $75\% \pm 5\%RH$ at $40^{\circ}C \pm 2^{\circ}C$, saturated salt solutions of NaBr and NaCl were prepared by dissolving the salts at $60^{\circ}C$. The slush of NaBr was kept at $25^{\circ}C \pm 2^{\circ}C$ and that of NaCl was kept at $40^{\circ}C \pm 2^{\circ}C$ in the dessicators. A dessicator with silica as a dessicant was also kept at $25^{\circ}C \pm 2^{\circ}C$ and vacuum was applied to this dessicator. The dessicators were allowed to equilibrate for 48 hours. A temperature humidity logger was kept in each dessicator for two days to record the temperature and humidity. The data obtained from these studies is shown in Figures 4, 5 and 6. Measurements were repeated monthly to ensure that the conditions prevailed throughout the stability studies when samples were in the dessicators.

Figure 4. Showing $25^{\circ}C \pm 2^{\circ}C$ and 0% humidity in the dessicator

STABILITY TESTING OF 900 µg OF LEVOTHYROXINE SODIUM (PURE DRUG)

Experimental Design: For these studies, 0.0009 gm (900 µg) of levothyroxine was carefully weighed and transferred to 4 ml HPLC vials. The vials were placed under different conditions in the dessicators. Three samples were withdrawn randomly from each dessicators at predetermined time intervals. HPLC analysis of these samples was performed to determine the percent of pure levothyroxine sodium left un-degraded. The studies were conducted for a period of 10 weeks.

Recovery Procedure: To a 4 ml HPLC vial containing 0.0009gm of a drug, 4ml of .01 M methanolic NaOH was added. The vials were shaken and kept in sonifier bath for 5 minutes, such that the drug dissolves in 4 ml of 0.01 M methanolic NaOH, to give an approximate concentration of 225 µg/ml. One ml of this solution was withdrawn and diluted in a 25 ml volumetric flask with 0.01 M methanolic NaOH, to get a concentration of 9 µg/ml. The samples were then analyzed by the HPLC method discussed earlier.

Note: The concentration of 9 µg /ml was selected to ensure that the sample concentration falls within the validated range of our HPLC method. Also, if the drug degrades over a period of time it would still be within the detection limits of the HPLC method.

RESULTS AND DISCUSSION:

The data presented in Table 9 shows the amount of drug remaining over a period of 10 weeks. Figure 7 shows a graphical presentation of the data. As can be seen from the plot, the drug appeared to be relatively stable at the different conditions evaluated except at 50°C. This is further corroborated by the fact that none of the kinetic models designed to

measure degradation fitted to the stability profiles of pure drug at the different conditions tested. It is believed that any trend in the degradation behavior of pure drug would have been accentuated and revealed by these models evaluated. Absence of any such trend within the limits of experimental variability coupled with the high values of percent drug left support this conclusion. Temperature induced degradation appeared to be significant at 50°C, as compared to the lower temperatures (see 25°C, 40°C and 50°C curves in Figure 7). These findings are supported by Won's study indicating a threshold temperature between 50°C and 60°C where levothyroxine sodium degrades rapidly [42]. Won, studied the kinetics of degradation of levothyroxine sodium in aqueous solution and in the solid state. The author concluded that levothyroxine sodium followed simple first-order degradation by the process of deiodination in aqueous solution. In contrast to solution degradation, levothyroxine sodium did not deiodinate in the solid state. Instead, the isolated degradation products

Table 9. Percentage of drug remaining after each time period under different conditions.

Time (Weeks)	25°C	25°C/60%RH	40°C/75%RH	50°C/20%moisture	50°C (dry)
Day 0	100.00	100.00	100.00	100.00	100.00
1	104.01	97.37	98.00	121.02	104.00
2	102.72	95.02	102.57	105.10	106.54
7	95.04	—	102.63	90.01	92.96
10	103.48	114.32	114.04	102.80	84.26

Figure 7. Percent levothyroxine remaining after storage for a period of 10 weeks.

indicated a deamination reaction. Furthermore, the solid-state degradation profiles showed biphasic first order degradation profiles, indicating the possibility of additional and complex degradation pathways [42].

As stated earlier, the drug exhibited stability at the different conditions monitored in this study. Studies at rather adverse conditions might have provided more insight into the degradation pathways. However, these are outside the scope of this study considering the FDA stipulated guidelines.

While the possibility of multiple complex degradation pathways may be indicated, what becomes important in context of predicting a model are those mechanisms that contribute significantly to that degradation. First order and biphasic first order models were evaluated to find the best fit for degradation kinetics of levothyroxine sodium under various conditions.

Statistical analysis of the data was performed using a non-linear curve fitting procedure from Sigma Plot for Windows (SPSS Inc., 1997). The raw data generated following the experimental procedures described earlier was evaluated using nonlinear regression. The goodness of fit of the experimental data for first-order and biphasic first-order model was evaluated using residual plots, adjusted r^2 values, normality testing etc. The equations used for first order and biphasic first order are shown below:

First-Order Equation

$$c = c_0 \cdot e^{-kt}$$

Biphasic First-Order Equation

$$c = a_1 e^{-k_1 t} + a_2 e^{-k_2 t}$$

Attempts to fit a model to understand the nature of degradation in this regime were unrewarding considering the stable behavior of pure drug. Inherent variability in the data arising as a result of the complex sample preparation (refer to page 40) and storage conditions involved further complicated the prediction of model. While no particular trend in the degradation profile of pure drug is seen at various conditions tested, it can be stated that the drug's stability seems unaffected at these conditions for short period of testing. An understanding of the stability behavior of pure levothyroxine sodium at the FDA stipulated conditions will form a basis for later studies which are expected to involve more complex multiple interactions.

III) DRUG-EXCIPIENT COMPATIBILITY TESTING

Drug-Excipient compatibility studies were conducted to determine the formulation ingredients and conditions that might provide a stable formulation of levothyroxine sodium.

Experimental Design: For these studies, nine commonly used excipients were selected. These excipients were dextrose, dicalcium phosphate dihydrate, calcium sulfate, mannitol, lactose anhydrous, lactose monohydrate, starch 1500, talc and ferric-oxide. The maximum strength of levothyroxine sodium tablets available in the market is 300 µg. According to FDA c-GMP guidelines, the sampling from the mixture should not be more than 3 times the maximum dose concentration available. Thus, it was decided to work at three times 900 µg. The levothyroxine sodium-excipient ratio was kept at 1:10.

Levothyroxine sodium and the excipients were mixed using a Crescent Wtg-L-Bug mixer. 0.0099gm of the mixture was carefully weighed out in separate 4 ml HPLC vials and recovery analysis was performed using the method described earlier. The vials were placed in the dessicators under the different conditions previously described. At defined time intervals, three samples for each drug-excipient mixture were analyzed. The studies were conducted for a period of 20 weeks.

RESULTS AND DISCUSSION:

The data presented in Tables 10 to 18 shows the amount of drug degraded in presence of different excipients over a period of 20 weeks. Figures 8 to 16 provide a graphical presentation of the data along with standard errors.

From the data obtained, it can be seen that levothyroxine sodium seems to be most stable in the presence of mannitol, starch 1500 and talc at 25°C, 25°C /60% RH and 40°C /75% RH. Drug degradation seems to be significant in the presence of some of the excipients containing carbohydrate. However, among this similar chemical class of excipients, wide differences in the degradation patterns were seen between dextrose and mannitol. While as little as 14% drug degradation was seen after 20 weeks in the presence of mannitol (40°C/75%RH condition), drug degradation was as large as 94% under similar experimental conditions in presence of the dextrose. In the order of increasing compatibility and stability of drug at 25°C, the various excipients evaluated can be ranked as: mannitol, starch 1500, ferric-oxide, talc, lactose hydrous, dicalcium phosphate dihydrate, dextrose, lactose anhydrous and calcium sulfate.

Table 10. Levothyroxine remaining in the presence of dextrose after each time period under different conditions.

Time(weeks)	25°C	25°C/60%RH	40°C/75%RH	50°C/20%moisture	50°C (dry)
Day 0	100.00	100.00	100.00	100.00	100.00
1	91.80	86.43	10.07	9.96	92.06
2	94.24	85.96	8.03	10.08	89.45
4	86.31	62.76	6.51	6.74	67.54
7	87.38	45.63	7.70	7.07	58.04
10	80.90	68.55	6.13	8.88	55.48
20	82.42	62.40	6.16	7.39	41.64

Dextrose

Figure 8. Levothyroxine remaining in the presence of dextrose over a period of 20 weeks.

Table 11. Levothyroxine remaining in the presence of dicalcium phosphate dihydrate after each time period under different conditions.

Time(weeks)	25°C	25°C/60%RH	40°C/75%RH	50°C/20%moisture	50°C (dry)
Day 0	100.00	100.00	100.00	100.00	100.00
1	95.65	70.89	76.94	92.90	66.97
2	78.56	84.03	71.82	93.12	77.15
3	121.70	74.15	98.00	103.12	57.96
4	78.53	78.06	87.40	91.35	78.81
7	65.81	78.11	67.55	70.38	59.33
10	93.03	72.84	68.22	67.38	44.61
20	87.00	125.68	62.91	56.59	43.44

Dicalcium Phosphate Dihydrate

Figure 9. Levothyroxine remaining in the presence of dicalcium phosphate dihydrate over a period of 20 weeks.

Table 12. Levothyroxine remaining in the presence of calcium sulfate after each time period under different conditions.

Time(weeks)	25°C	25°C/60%RH	40°C/75%RH	50°C/20%moisture	50°C (dry)
Day 0	100.00	100.00	100.00	100.00	100.00
1	91.92	88.39	86.92	74.72	84.16
2	90.09	86.84	84.26	82.37	83.91
3	91.00	89.35	87.23	76.19	81.98
4	83.74	87.87	85.57	78.41	72.51
7	83.69	88.73	78.27	89.02	60.83
10	82.41	90.89	85.30	68.50	60.80
20	74.80	80.26	83.28	47.34	54.93

Calcium Sulfate

Figure 10. Levothyroxine remaining in the presence of calcium sulfate over a period of 20 weeks.

Table 13. Levothyroxine remaining in the presence of mannitol after each time period under different conditions.

Time(weeks)	25°C	25°C/60%RH	40°C/75%RH	50°C/20%moisture	50°C (dry)
Day 0	100.00	100.00	100.00	100.00	100.00
1	99.62	96.91	96.84	100.78	95.69
2	101.32	100.96	97.75	95.21	96.09
3	99.32	97.14	92.61	90.57	91.04
4	96.46	91.51	94.59	91.03	89.51
7	94.71	92.11	92.13	87.26	59.09
10	89.22	94.78	98.23	92.47	75.06
20	91.85	98.42	86.41	83.05	70.70

Mannitol

Figure 11. Levothyroxine remaining in the presence of mannitol over a period of 20 weeks.

Table 14. Levothyroxine remaining in the presence of lactose anhydrous after each time period under different conditions.

Time(weeks)	25°C	25°C/60%RH	40°C/75%RH	50°C/20%moisture	50°C (dry)
Day 0	100.00	100.00	100.00	100.00	100.00
1	91.87	89.59	78.30	75.98	88.87
2	91.61	91.36	76.57	61.55	66.12
3	88.27	82.51	76.78	58.09	64.97
4	84.40	81.11	70.83	54.74	81.79
7	74.94	69.22	63.14	58.91	43.14
10	73.66	69.21	63.51	53.14	45.48
20	66.07	61.79	56.62	50.94	43.87

Lactose Anhydrous

Figure 12. Levothyroxine remaining in the presence of lactose anhydrous over a period of 20 weeks.

Table 15. Levothyroxine remaining in the presence of lactose hydrous after each time period under different conditions.

Time(weeks)	25°C	25°C/60%RH	40°C/75%RH	50°C/20%moisture	50°C (dry)
Day 0	100.00	100.00	100.00	100.00	100.00
1	96.86	93.86	83.35	75.17	96.77
2	98.54	97.16	79.34	61.08	85.06
3	101.98	86.79	77.71	59.80	75.39
4	84.23	83.68	80.61	65.94	67.96
7	86.59	85.21	69.87	53.29	65.11
10	83.88	69.05	71.35	60.15	56.60
20	76.69	79.65	67.43	34.35	37.29

Lactose Hydrous

Figure 13. Levothyroxine remaining in the presence of lactose hydrous over a period of 20 weeks.

Table 16. Levothyroxine remaining in the presence of starch 1500 after each time period under different conditions.

Time(weeks)	25°C	25°C/60%RH	40°C/75%RH	50°C/20%moisture	50°C (dry)
Day 0	100.00	100.00	100.00	100.00	100.00
1	100.39	101.70	100.85	97.26	94.65
2	99.84	103.89	103.37	90.47	89.57
3	95.66	97.61	99.91	86.07	87.21
4	96.92	96.50	101.55	80.36	76.05
6	95.74	96.69	98.80	64.39	63.08
8	92.10	95.93	102.38	68.50	59.58
10	96.53	98.30	100.66	72.19	53.85
20	88.98	93.90	99.50	73.78	41.01

Starch 1500

Figure 14. Levothyroxine remaining in the presence of starch 1500 over a period of 20 weeks.

Table 17. Levothyroxine remaining in the presence of talc after each time period under different conditions.

Time(weeks)	25°C	25°C/60%RH	40°C/75%RH	50°C/20%moisture	50°C (dry)
Day 0	100.00	100.00	100.00	100.00	100.00
1	103.49	102.67	99.12	87.19	100.14
2	103.05	99.86	100.06	79.11	87.20
3	97.36	98.22	97.43	66.64	79.61
4	95.07	101.49	104.43	73.49	77.79
6	96.59	101.99	100.10	64.01	68.88
8	88.43	98.32	108.48	68.72	64.40
10	89.71	94.77	98.60	58.90	60.01
20	80.15	94.11	99.70	59.92	39.45

Talc

Figure 15. Levothyroxine remaining in the presence of talc over a period of 20 weeks.

Table 18. Levothyroxine remaining in the presence of ferric-oxide after each time Period under different conditions.

Time(weeks)	25°C	25°C/60%RH	40°C/75%RH	50°C/20%moisture	50°C (dry)
Day 0	100.00	100.00	100.00	100.00	100.00
1	90.09	93.16	94.61	85.05	87.89
2	97.45	96.88	97.73	91.40	87.90
3	93.80	91.54	99.21	97.04	83.48
4	93.10	89.95	97.38	88.44	73.69
6	91.25	94.38	93.34	84.27	70.19
8	85.47	88.63	88.69	84.95	62.02
10	84.21	89.28	88.46	85.17	60.72
20	86.16	91.71	94.17	85.21	45.03

Ferric Oxide

Figure 16. Levothyroxine remaining in the presence of ferric-oxide over a period of 20 weeks.

Considering that a Schiff-base reaction and an oxidation reaction may be the predominant pathways [43, 46], first-order and biphasic first-order models were evaluated to find the best fit for degradation kinetics of levothyroxine sodium in the presence of different excipients under various conditions. Significant degradation involving any single pathway is best explained by simple first order kinetics as seen with dextrose, where Schiff-Base reaction is seemingly predominant. On the other hand, degradation following two different mechanisms (Schiff-base reaction plus oxidation) that are regulated by different rate kinetics seems to be best explained by bi-phasic first order models. The majority of the drug-excipient mixtures that were evaluated exhibited dual mechanism as reflected in the number of successfully fitted biphasic first order models. A non-linear regression analysis similar to the one used for the pure drug data was performed on the experimental data obtained from drug-excipient compatibility studies. The complete output of this statistical analysis along with the model parameters and constraints are shown in detail in Appendix A. Residual plots are generated from this statistical output using suitable graphing procedure (Microsoft® Excel 97) and are shown in Appendix B. After selecting the model that fits the data following the above analysis, expiration dates (t_{90}) for levothyroxine sodium in the presence of different excipients were calculated. For all conditions, the k -values for first order reactions and a_1 , a_2 , k_1 and k_2 for biphasic first order reactions were calculated from the models and used to calculate t_{90} values. The summary of the various results obtained is presented in Table 19.

It was noticed that levothyroxine degraded to a higher extent at 50°C in presence of all the excipients. These findings further support the fact that 50°C- 60°C is the threshold

Table 19. T₉₀ values for different drug-excipient mixtures at different conditions.

Excipients	t-90 in Weeks				
	25 C	25 C/ 60% RH	40 C/ 75 % RH	50 C/ 20% moisture	50 C
Dextrose	2.77	0.76	0.001	5.29E-06	1.23
DiCalcium Phosphate Dihydrate	2.98	< 1	0.54	3.18	2.38
Calcium Sulfate	1.89	16.4	0.56	0.023	0.94
Mannitol	24.22	153.6	19.44	6.26	2.29
Lactose Anhydrous	2.13	1.55	0.29	0.29	0.68
Lactose Hydrous	7.29	5.1	2.38	0.22	0.45
Starch	18.1	28.37	> 20	1.61	1.67
Talc	9.24	26.92	> 20	0.68	1.67
Ferric Oxide	14	153.6	307.2	2.74	1.26

temperature for the degradation of levothyroxine sodium. Among all of the different types of excipients used, degradation of levothyroxine sodium occurs most quickly in the presence of carbohydrates such as dextrose, lactose hydrous, lactose anhydrous and starch. At higher temperatures, it is theorized that the ring structure of the carbohydrates breaks to open up a free aldehyde group. This leads to a Schiff-base reaction between a

free aldehyde group of the carbohydrates and the highly reactive amino group of levothyroxine sodium. The general Schiff-base reaction is shown in Figure 17.

Figure 17. Reaction mechanism for Schiff-base reaction

The availability of the free aldehyde group varies among the carbohydrates. For example, dextrose which is composed of only one ring structure, can easily break open to give a free aldehyde group, and so by this mechanism the drug would degrade very quickly in presence of dextrose. Lactose, which is composed of a two-ring structure, would be more stable with the drug as compared to dextrose but less stable as compared to starch. Levothyroxine sodium was found to be much more stable in presence of mannitol. This could be attributed to the absence of the ring structure and the free aldehyde group in mannitol.

The literature also indicates that levothyroxine has a tendency to undergo oxidation reaction [43]. Accordingly, in the presence of various components capable of oxidizing the pure drug, the degradation can be expected to be rather severe. It was seen that the mixture of levothyroxine with carbohydrates had turned brown, and this may be attributed to the oxidation of the drug by the mechanism shown in Figure 18.

Figure 18. Reaction mechanism for Oxidation

The overall results showed that the drug was most stable at 25°C and 25°C / 60% RH. One of the most interesting findings observed was in the case of talc and starch, where levothyroxine sodium was much more stable at 25°C / 60% RH and 40°C / 75% RH in presence of these excipients (Figure 14 and 15) than at the same temperatures with added humidity. This behavior may be due to a higher moisture uptake ability of starch and talc as compared to other excipients [47]. A higher affinity of these excipients towards moisture might have sequestered the free moisture, thereby reducing the availability of water for reaction with pure drug.

Overall for this group of excipients the lowest t-90 values of levothyroxine sodium were observed in the presence of dextrose, dicalcium phosphate dihydrate and calcium sulfate at 25°C, 25°C / 60% RH and 40°C / 75% RH (Table 19) with stability well below usable levels.

Interestingly, the stability of the mixtures of levothyroxine sodium and lactose anhydrous were much lower as compared to that of levothyroxine sodium and lactose monohydrate as seen in Table 19 (Figures 23 and 24). HPLC analysis demonstrated that at higher humidity/temperature, the mixture containing lactose anhydrous exhibited relatively greater degradation of the drug than that containing lactose monohydrate. This type of degradation was reported by Jain et al. [29], who studied the stability of a proprietary hydrophobic drug in the presence of hydrous and anhydrous lactose. The authors concluded that, lactose anhydrous becomes hydrated on exposure to high humidity/temperature condition and that the transition state of the lactose, not its stable state may be responsible for its greater interaction and subsequent degradation of the drug. They also concluded that in certain cases, lactose anhydrous may absorb a

significant amount of moisture, which can affect its inherent properties and may directly come in contact with the drug. For a moisture sensitive drug like levothyroxine sodium, this behavior may drastically affect its stability. Therefore, the general belief that lactose anhydrous, which has less than 0.5% moisture, should provide greater stability as compared to lactose hydrous needs to be properly evaluated.

The comparative effects of the different storage conditions on the stability of each of the drug-excipient mixtures are presented in Figures 19 to 27.

Figure 19. Effect of storage conditions on drug+dextrose mixture

Figure 20. Effect of storage conditions on drug+DCPD mixture

Figure 21. Effect of storage conditions on drug+calcium sulfate mixture

Figure 22. Effect of storage conditions on drug+mannitol mixture

Figure 23. Effect of storage conditions on drug+lac.anhydrous mixture

Figure 24. Effect of storage conditions on drug+lac.hydrus mixture

Figure 25. Effect of storage conditions on drug+starch 1500 mixture

Figure 26. Effect of storage conditions on drug+talc mixture

Figure 27. Effect of storage conditions on drug+ferric-oxide mixture

IV) LEVOTHYROXINE SODIUM TABLETS AND PHYSICAL TESTS

A) FORMULATION OF LEVOTHYROXINE SODIUM TABLETS

After the completion of drug-excipient compatibility studies, evaluation of the stability of tablet formulations proceeds in two consecutive steps; the first is the formulation of tablets that meet USP criteria and second is to monitor stability under various conditions. The present study is designed to formulate and evaluate the properties of levothyroxine tablets.

METHODS AND MATERIALS:

Materials:

Sodium Hydroxide (Fisher Scientific, Lot # S318-500), Methanol (HPLC Grade, Fisher Scientific, Lot # A452-4), Acetonitrile (HPLC Grade, Fisher Scientific, Lot # A998-4), O-Phosphoric Acid (Fisher Scientific, Lot # A260-500), Mannitol (SPI Polyols, Inc. Lot # 3127G9), Starch 1500 (National Starch and Chemical Company, Lot # CB7137), Talc (Cyprus Industrial Minerals Co., ID # ACM-189-7-03), Deionized water, pH/mv meter (model 420A, Orion Research, Inc.), Stokes Single Punch Tablet Press, Turbula mixer, Tablet Hardness Tester, USP Dissolution Apparatus, USP Disintegration Apparatus, HPLC System (Waters, Inc.), Magnetic Stirrer (Thermix® Model 120 MR), Weighing balance (Metler, Model # AE 240) and Sieve # 40.

EXPERIMENTAL PROCEDURE:

Based on the results obtained from drug-excipient compatibility studies, the more stable excipients, mannitol(diluent), starch 1500(disintegrant and binder) and talc(lubricant)

were selected for use in formulating the tablets. Due to the low concentration of the levothyroxine sodium in the tablets drug-to-excipient ratios of the excipients slightly different from those studied in the compatibility studies were used to obtain tablets of consistent weight and hardness. The total weight of the tablets was fixed at 115mg. Table 20 indicates the selected formulation.

Table 20. Composition of levothyroxine sodium tablet formulation

Ingredients	Composition (%)	Composition (mg/tablet)	Batch of 30,000 tablets (gms)
Levothyroxine Sodium	0.299	0.345	0.9
Mannitol	93.718	108.1	282
Starch 1500	4.985	5.75	15
Talc	0.997	1.15	3

Tabletting Procedure

Ingredients for the batch of 30,000 tablets, each weighing 115mg and containing 345µg of levothyroxine sodium were weighed as shown in Table 20.

All the excipients as well as the drug were passed through a #40 sieve to get uniform particle size distribution. 282 gm of mannitol, 15 gm of starch 1500 and 3 gm of talc were weighed into a jar and mixed for 5 minutes using Turbula® mixer. Levothyroxine sodium was added to the remaining ingredients using geometric dilution while mixing. The overall optimum mixing time was estimated to be 20 minutes. This was determined by addition of a red dye to the excipient mixture and observation of the homogeneity of the dye dispersed in the mixture at various time intervals. Further to confirm the homogeneity of the mixture, a 100mg sample of the powder was taken from 6 different points in the container and compared with standard L-4 (6mcg/ml). Results of homogeneity testing are shown in Table 21. The amount of drug recovered expressed as

mean (% CV) of six random samples was found to be 98.6% (7.00). Accordingly, the homogeneity of the drug mixture passes the USP specified limits (85% – 115%).

Table 21. Homogeneity check for 100mg of powder mixture as compared to standard (6 ug/ml) after 20 minutes of mixing.

	Mean % Recovery
Sample 1	103.78
Sample 2	96.32
Sample 3	109.03
Sample 4	98.36
Sample 5	94.51
Sample 6	89.61
<i>Mean</i>	98.6
<i>Std. Dev</i>	6.9
<i>% CV</i>	7

An ANOVA was applied to the data and the p-value of .07 obtained indicated no significant difference between these samples. The mixture was considered acceptable and tablets were compressed using a Stokes® single punch tableting machine. The following tests were performed to check the general properties of the tablets:

- 1) **Weight Variation:** Twenty tablets were selected at random and weighed. The weight of these tablets are shown in Table 22.

USP limits: For tablet weights below 130mg, percentage difference accepted is $\pm 10\%$ which allows weight limits for these tablets of 105mg to 125mg.

Average weight of 20 tablets = 116.05mg

Allowable deviation is 10%

Number of tablets in sample exceeding limits was 0 tablets

Result: Therefore the batch passes the weight variation test

Table 22. Weight of 20 tablets for weight variation test

	Tablet Weight (gm)		Tablet Weight (gm)
Tablet 1	0.123	Tablet 11	0.122
Tablet 2	0.114	Tablet 12	0.113
Tablet 3	0.114	Tablet 13	0.107
Tablet 4	0.119	Tablet 14	0.110
Tablet 5	0.117	Tablet 15	0.113
Tablet 6	0.115	Tablet 16	0.122
Tablet 7	0.111	Tablet 17	0.123
Tablet 8	0.120	Tablet 18	0.120
Tablet 9	0.107	Tablet 19	0.118
Tablet 10	0.121	Tablet 20	0.112

- 2) Content Uniformity: 30 Tablets were selected at random, out of which 10 tablets were randomly chosen for the content uniformity test. The data for content uniformity test is presented in Table 23.

USP Limits: The concentration of the drug should lie in the range of 85% to 115% and the %CV should be less than or equal to 6.0%.

Results: The tablets passed the USP content uniformity test.

- 3) Tablet Hardness: Hardness of 10 tablets was determined using the Erweka® tablet hardness tester and the data is shown in Table 24. The average hardness of the tablets was 7.125kg.

Table 23. Data for content uniformity test for 115mg tablets

	% of L-4
Tablet 1	96.03
Tablet 2	109.3
Tablet 3	105.82
Tablet 4	107.07
Tablet 5	101.21
Tablet 6	99.89
Tablet 7	97.87
Tablet 8	93.28
Tablet 9	103.04
Tablet 10	90.95
<i>Mean % Recovery</i>	100.45
<i>Std. Dev</i>	6.02
<i>% CV</i>	5.87

Table 24. Data for hardness test for levothyroxine tablets

	Hardness in kg
Tablet 1	7.00
Tablet 2	6.50
Tablet 3	7.50
Tablet 4	7.00
Tablet 5	7.50
Tablet 6	7.25
Tablet 7	6.75
Tablet 8	7.50
Tablet 9	7.50
Tablet 10	6.75
<i>Mean</i>	7.13
<i>Std. Dev</i>	0.38
<i>% CV</i>	5.30

4) Friability Test:

Weight of 10 tablets = 1.161gms

Weight of 10 tablets after 150 revolutions (5minutes) = 1.153gms

Percentage weight loss of 10 tablets = 0.69%.

USP Limits: A maximum weight loss of not more than 1% of the weight of tablets being tested is considered acceptable for most products.

Results: The tablets passed USP friability test.

- 5) Disintegration Test: The USP (Vanderkamp) disintegration tester was used for these studies. 1000 ml of distilled water was placed in a glass beaker and was kept in the water bath maintained at the temperature $37 \pm 2^{\circ}\text{C}$. Six tablets were selected and the disintegration time noted. All tablets disintegrated within 5 minutes.

USP Limits: All six tablets should disintegrate within 30 minutes.

Results: The tablets passed the USP disintegration test.

- 6) Dissolution Test: USP apparatus 2 was used. The apparatus was set to 100 rpm. The temperature of water bath was set to $37 \pm 2^{\circ}\text{C}$.

Dissolution media: .05M phosphate buffer with pH 7.4; 500ml.

Dissolution Studies: 500 ml of pH 7.4, .05M phosphate buffer was filled in the dissolution flasks and the system was allowed to equilibrate for 30 minutes. Six tablets weighing 105mg were selected and placed in each of the six dissolution flasks. Sampling was performed at predetermined time points, namely 10, 20, 30, 45, 60, 80 and 100 minutes. At each time interval, 5 ml of the sample was withdrawn from the dissolution flask and 1 drop of phosphoric acid was added to

the sample. The media was not replaced because the volume of dissolution media after sampling was still adequate to maintain sink conditions (solubility of L-4 in phosphate buffer pH 7.4 is .022 - .044 gm/100ml) [43]. A correction factor was used to correct for the volume of the media not replaced, and hence to calculate the percent of drug dissolved. The samples were directly analyzed by HPLC. Table 25 shows the data obtained. Figure 28 shows the dissolution profile of levothyroxine sodium from its tablets over the 100 minutes of run time.

Results: USP limits state that not less than 55% (*Q*) of the labeled amount of Levothyroxine sodium is dissolved in 80 minutes. Hence the tablets passed the dissolution test.

Table 25. Dissolution of levothyroxine sodium tablets

Time (minutes)	Mean % Dissolved n=6	Standard Deviation
0	0	0
10	79.32	11.05
20	82.22	4.99
30	74.83	2.79
45	79.82	8.83
60	76.87	4.1
80	78.58	8.87
100	74.86	6.6

Figure 28. Dissolution profile of levothyroxine sodium in tablet formulation

B) EVALUATION OF THE STABILITY OF LEVOTHYROXINE SODIUM TABLETS

Evaluation of the stability of formulation is an integral part of the development process. A complete understanding of the stability of the formulation can only be obtained after the underlying component interactions are studied. This also helps the formulator in selecting the tableting excipients that should offer optimum stability. The importance of such studies is exemplified by cases like the marketed levothyroxine sodium formulations where the potency could not be assured through the expiration date. The many problems and issues associated with levothyroxine sodium formulations have been discussed in the earlier section (Chapter 1).

In view of the above facts, studies were performed to evaluate the stability of pure drug and excipient compatibility. Based on these results an optimum formulation components were selected. Further tablet testing was performed on the selected formulation and the tablets produced were found to conform to the USP standards. This portion of work deals with the stability testing of these tablets.

Experimental Design:

The previously formulated and manufactured levothyroxine sodium tablets were stored in stability chambers under same conditions as excipients. The stability of the tablets was evaluated over a period of 10 weeks. Tablets were sampled at predetermined time points and drug content was determined after extraction using the previously discussed HPLC method. At the end of 10 weeks, dissolution studies were also conducted on these tablets, that had been stored at 25°C / 60%RH and at 40°C / 75%RH to determine if dissolution was affected by moisture.

RESULTS AND DISCUSSION: From the results obtained from the drug-excipient compatibility studies, it was decided to use mannitol (diluent), starch 1500 (binder and disintegrant) and talc (lubricant) as the excipients in the formulation of the levothyroxine sodium tablets. Immediately after manufacture, the tablets were characterized by good mechanical strength and a shorter disintegration time than the limits specified in the USP. Further dissolution studies indicated that 80% of levothyroxine sodium was released within 30 minutes. The data for stability studies after 10 weeks is shown in table 26 and Figure 29.

From the data (Table 26) it can be seen that the drug degraded to less than 90% in less than a week, at all conditions studied. The stability of tablets was found to be much worse than the stability of levothyroxine sodium determined in presence of individual excipients (refer to Figure 29 versus Figures 11, 14 and 15). It appears that the rate of degradation of levothyroxine sodium contained in the tablets was the synergistic effect of the individual excipients used. This is reflected in the shape of the degradation curves for the tablets when compared to those of individual excipients. A general trend showing an initial faster degradation rate followed by a relatively slower phase is apparent for all the conditions studied.

The results of the dissolution studies of the tablets stored for 10 weeks are presented in Table 27 and 28 and Figures 30 and 31. The amount of drug released was corrected basing on the percentage of drug left undegraded under respective conditions. Dissolution profiles were found to be identical when compared to the freshly manufactured tablets. Basing on these findings it could be stated that the physical stability of the formulation did not change at the end of 10 weeks. Dissolution testing of tablets is considered as a

quality-indicating tool in ensuring batch to batch uniformity [2]. For our purposes, it is therefore believed that any minor changes in the physical stability of the formulation would have been accentuated and reflected in the dissolution behavior of tablets.

Table 26. Evaluation of the stability of levothyroxine sodium tablets.

Time(weeks)	25°C	25°C/60%RH	40°C/75%RH	50°C (dry)
Day 0	100.00	100.00	100.00	100.00
1	83.25	83.46	91.45	73.73
2	74.83	82.42	82.64	75.25
7	81.68	94.57	72.52	54.66
10	77.50	79.37	80.88	52.14

Figure 29. Stability profile of levothyroxine sodium tablets

Table 27. Dissolution studies of levothyroxine sodium tablets after 10 weeks at 25°C / 60%RH.

Time (minutes)	Mean % Dissolved n=6
0	0
10	87.88
20	97.24
40	90.73
60	95.54
80	93.21
100	92.89

Table 28. Dissolution studies of levothyroxine sodium tablets after 10 weeks at 40°C / 75%RH.

Time (minutes)	Mean % Dissolved n=6
0	0.00
10	32.10
20	44.85
40	66.16
60	78.78
80	87.35
100	79.91

Figure 30. Dissolution profile of levothyroxine sodium tablets stored at 25°C / 60%RH after 10 weeks

Figure 31. Dissolution profile for levothyroxine sodium tablets stored at 40°C / 75%RH after 10 weeks

To better define the rate of degradation of the tablets as compared to the excipients, first-order and biphasic first-order models were evaluated using Sigma Plot. Details of the modeling procedure have been discussed earlier (refer to page 43). Results of the statistical analysis and goodness of fit calculations are compiled in appendices A and B. T_{90} values at the different storage conditions studied were derived from the kinetic parameters obtained from best fit models. In few cases of excipient/conditions (DCPD and levothyroxine sodium tablets at 25°C/60%RH and starch and talc at 40°C/75%RH) limited success was seen in fitting a kinetic model for degradation. A combination of factors such as the complexity in degradation, variability in the data etc., restricted selection of the best kinetic model.

For comparative purposes, the effect of series of storage conditions on t_{90} values of levothyroxine in tablets are shown together with those of single excipient mixtures in Figures 32 to 35. As can be seen from Figures 32 to 35, the t_{90} values of levothyroxine sodium tablets are significantly lower when compared to those determined in the presence of individual excipients. As discussed earlier the excipients that offered the best t_{90} values and those exhibiting the highest compatibility on an individual basis were selected for the final formulation. However, it can be seen that in combination, the incompatibility caused by these excipients was more seemingly complex. For example, the t_{90} values of levothyroxine sodium at 25°C in tablets dropped by 98.55% when compared to that in presence of pure mannitol. As a result of the rapid degradation seen in presence of the tablet formulation at all storage conditions studied no particular trend was observed. The rapid rate of degradation is believed to mask the effect of temperature and humidity on the final formulation.

Figure 32. T_{90} of levothyroxine sodium in presence of different excipients and as a tablet when stored at 25°C

*t-90 for DCPD and L-4 tablets * is < 1 week*

Figure 33. T_{90} of levothyroxine sodium in presence of different excipients and as a tablet when stored at 25°C / 60% RH

t-90 for Starch and Talc* is > 20 week*

Figure 34. T_{90} of levothyroxine sodium in presence of different excipients and as a tablet when stored at 40°C / 75% RH

Figure 35. T₉₀ of levothyroxine sodium in presence of different excipients and as a tablet when stored at 50°C

Figure 36 shows that estimated t_{90} values for the tablet formulation under all storage conditions studied were found to be less than one week. The loss of potency often observed in the marketed levothyroxine sodium formulations was confirmed by these results.

It remains to be seen why levothyroxine sodium in tablets exhibited a complex and rapid degradation as compared to pure levothyroxine sodium and levothyroxine sodium in presence of single excipients. Compatibility studies involving multiple excipients and evaluation of the effect of environmental conditions during manufacture may help in understanding this behavior. To this objective, our studies are believed to form a firm basis for future research in this direction.

t-90 for L-4 tablets at 25 C/ 60 %RH is < 1 week

t-90 for L-4 tablets at 50 C is 3.3 E-05

Figure 36. Effect of storage conditions on levothyroxine sodium tablets

V) SUMMARY AND CONCLUSIONS

The proposed testing program was designed to evaluate excipient compatibility studies with levothyroxine sodium as a pre-formulation tool. The results provided an insight on the stability of the drug in the presence of single as well as multiple excipients under the FDA stipulated conditions of temperature and humidity.

The acquired results indicated that the pure drug was relatively stable for up to 10 weeks under different conditions evaluated except at 50°C. Won CM [42] had observed rapid degradation of the pure drug above 50°C. A similar trend was observed in our studies, which indicates that there might be additional and complex degradation pathways at higher temperatures.

Nine commonly used excipients were selected for the compatibility studies. These excipients were dextrose, dicalcium phosphate dihydrate, calcium sulfate, mannitol, lactose anhydrous, lactose monohydrate, starch 1500, talc and ferric-oxide. In the presence of these excipients the stability of the drug decreased as compared to that of pure drug with the extent of degradation dependant on the chemical properties of the excipients. The lowest drug degradation of the investigated drug-excipient mixtures was observed in the drug mixtures with mannitol, starch 1500 and talc. Considerable degradation of the drug was noted in the presence of carbohydrates having an aldehyde group. This behavior of the drug in presence of carbohydrates was attributed to Schiff-base and oxidation reactions. The lowest t-90 values for the drug was observed in the presence of dextrose, dicalcium phosphate dihydrate and calcium sulfate at 25°C, 25°C / 60% RH and 40°C / 75% RH (Table 19) with stability well below usable levels.

The results of the compatibility studies led to the selection of mannitol, starch 1500 and talc for use in formulating levothyroxine sodium tablets. The formulated tablets were found to conform to USP standards. The data on the stability of tablets revealed that the drug degraded to less than 90% in less than a week at all the conditions studied. These results indicate that in the presence of multiple excipients, the stability of the drug appeared to be much less. No particular trend was observed in the degradation profile as a function of temperature and humidity on the final formulation. It appears that the rapid degradation of the drug in tablets may be due to the synergistic effect of the individual excipients used or the processing variables such as mixing techniques, compaction pressures etc.

From the overall results obtained it can be concluded that levothyroxine sodium has very little stability at 50°C. It is also recommended that dextrose, lactose hydrous, lactose anhydrous, calcium sulfate and dicalcium phosphate dihydrate should not be used as excipients along with levothyroxine due to the very low stability in presence of these excipients.

VI) RECOMMENDATIONS FOR FUTURE WORK

The future work on the stability studies of levothyroxine sodium should focus on the following:

- Increase the drug concentration and study the degradation pattern using IR, NMR or DSC for better understanding of the degradation pathways.
- Investigate the stability of the drug in presence of different formulation environments like adjusting micro-environmental pH or addition of anti-oxidants

or coating the drug particles with inert polymers and thereby reducing the contact of the drug with excipients.

- Stability of the drug in presence of different mini-formulations (multiple excipients) consisting of different compositions and ratios.
- Evaluate the effect of processing conditions and equipments on the stability of the drug.

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APPENDIX A

INTRODUCTION:

This appendix includes the statistical output of non-linear regression for first-order and biphasic first-order reactions. Sigma plot for Windows (Version 4, SPSS Inc., 1997) was used to perform the curve fitting procedures for both the models. First section gives the definition of the variables, constraints and model parameters. This is followed by tests to validate the equal variance and normality assumptions for model errors. The final section gives the residuals and other diagnostics. Salient parameters basing on which out conclusions are made, include adjusted r^2 values, normality tests and residual plots indicating the goodness of the fit followed by providing the validity of the assumptions.

DEXTROSE (First-order reaction)

25°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 94.074}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.00908564}}

[Equation]

f=a*exp(-b*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

[Options]

tolerance=0.0001

stepsize=100

iterations=100

R = 0.79403850 Rsqr = 0.63049713 Adj Rsqr = 0.55659656

Standard Error of Estimate = 4.5081

	Coefficient		Std. Error	t	P
a	94.0740	2.4474	38.4386	<0.0001	
b	0.0091	0.0032	2.8696	0.0350	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	173.3889	173.3889	8.5317	0.0330
Residual	5	101.6146	20.3229		
Total	6	275.0034	45.8339		

PRESS = 385.8371

Durbin-Watson Statistic = 2.1014

Normality Test: Passed (P = 0.7083)

Constant Variance Test: Passed (P = 0.7810)

Power of performed test with alpha = 0.0500: 0.5810

The power of the performed test (0.5810) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	94.0740	5.9260	1.3145	1.5653	1.9604
2	93.2232	-1.4185	-0.3147	-0.3623	-0.3284
3	92.3800	1.8564	0.4118	0.4625	0.4228
4	90.7165	-4.4046	-0.9770	-1.0648	-1.0830
5	88.2772	-0.9019	-0.2001	-0.2167	-0.1948
6	85.9036	-5.0004	-1.1092	-1.2432	-1.3378
7	78.4427	3.9775	0.8823	1.7381	2.4711

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFBETS
1	0.5119	0.2947	1.2673
2	0.0214	0.2459	-0.1876
3	0.0279	0.2072	0.2161
4	0.1064	0.1580	-0.4691
5	0.0041	0.1479	-0.0811
6	0.1980	0.2040	-0.6771
7	4.3516	0.7423	4.1943

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	94.0740	87.7828	100.3652	80.8880	107.2600
2	93.2232	87.4765	98.9698	80.2881	106.1582
3	92.3800	87.1052	97.6548	79.6476	105.1124
4	90.7165	86.1103	95.3227	78.2462	103.1868
5	88.2772	83.8206	92.7339	75.8614	100.6931
6	85.9036	80.6701	91.1370	73.1882	98.6189
7	78.4427	68.4584	88.4271	63.1464	93.7391

2D Graph 2

***** DEXTROSE (First-order reaction)

25°C / 60 %RH

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 85.5238}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.0276726}}

[Equation]

f=a*exp(-b*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

[Options]

tolerance=0.0001

stepsize=100

iterations=100

R = 0.63120454

Rsqr = 0.39841917

Adj Rsqr = 0.27810301

Standard Error of Estimate = 15.7556

	Coefficient		Std. Error	t	P
a	85.5238	9.0764	9.4226	0.0002	
b	0.0277	0.0157	1.7668	0.1375	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	822.0289	822.0289	3.3114	0.1285
Residual	5	1241.1974	248.2395		
Total	6	2063.2264	343.8711		

PRESS = 3064.4031

Durbin-Watson Statistic = 1.2174

Normality Test: Passed (P = 0.0576)

Constant Variance Test: Passed (P = 0.9684)

Power of performed test with alpha = 0.0500: 0.3181

The power of the performed test (0.3181) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	85.5238	14.4762	0.9188	1.1241	1.1630
2	83.1896	3.2403	0.2057	0.2392	0.2151
3	80.9191	5.0445	0.3202	0.3599	0.3262
4	76.5623	-13.8018	-0.8760	-0.9526	-0.9417
5	70.4629	-24.8293	-1.5759	-1.7223	-2.4156
6	64.8494	3.6990	0.2348	0.2693	0.2426
7	49.1729	13.2297	0.8397	1.4033	1.6122

Influence Diagnostics:

Row	Cook's Dist		Leverage	DFFITS
1	0.3138	0.3319	0.8197	
2	0.0101	0.2605	0.1277	
3	0.0171	0.2088	0.1676	
4	0.0828	0.1543	-0.4022	
5	0.2885	0.1628	-1.0653	
6	0.0114	0.2398	0.1363	
7	1.7657	0.6420	2.1589	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	85.5238	62.1921	108.8555	38.7830	132.2646
2	83.1896	62.5191	103.8600	37.7186	128.6605
3	80.9191	62.4128	99.4253	36.3902	125.4479
4	76.5623	60.6541	92.4704	33.0489	120.0756
5	70.4629	54.1204	86.8054	26.7889	114.1369
6	64.8494	45.0165	84.6824	19.7531	109.9458
7	49.1729	16.7218	81.6239	-2.7252	101.0709

2D Graph 3

..... **DEXTROSE (First-order reaction)**

40°C / 75 %RH

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 99.9044}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 2.14682}}

[Equation]

f=a*exp(-b*x)

fit f to y
 "fit f to y with weight reciprocal_y
 "fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

[Options]

tolerance=0.0001

stepsize=100

iterations=100

R = 0.98465494 Rsqr = 0.96954536 Adj Rsqr = 0.96345443

Standard Error of Estimate = 6.6937

	Coefficient		Std. Error	t	P
a	99.9044	6.6931	14.9265	<0.0001	
b	2.1468	0.5596	3.8362	0.0122	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	7132.0364	7132.0364	159.1786	<0.0001
Residual	5	224.0263	44.8053		
Total	6	7356.0627	1226.0105		

PRESS = 282946.1962

Durbin-Watson Statistic = 0.3366

Normality Test: Failed (P = 0.0025)

Constant Variance Test: Passed (P = 0.2965)

Power of performed test with alpha = 0.0500: 0.9981

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	99.9044	0.0956	0.0143	1.0642	1.0823
2	11.6744	-1.6080	-0.2402	-1.0510	-1.0650
3	1.3642	6.6708	0.9966	1.0238	1.0300
4	0.0186	6.4913	0.9698	0.9698	0.9627
5	0.0000	7.7013	1.1505	1.1505	1.2001
6	0.0000	6.1283	0.9155	0.9155	0.8976
7	0.0000	6.1587	0.9201	0.9201	0.9029

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	3143.8469 0.9998	80.6421	
2	10.0185 0.9478	-4.5360	
3	0.0290 0.0524	0.2422	
4	0.0000 0.0000	0.0061	
5	0.0000 0.0000	0.0000	
6	0.0000 0.0000	0.0000	
7	0.0000 0.0000	0.0000	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	99.9044	82.6993	117.1095	75.5716	124.2372
2	11.6744	-5.0767	28.4255	-12.3395	35.6883
3	1.3642	-2.5741	5.3025	-16.2874	19.0158
4	0.0186	-0.0901	0.1274	-17.1883	17.2256
5	0.0000	-0.0003	0.0003	-17.2066	17.2067
6	0.0000	-0.0000	0.0000	-17.2066	17.2066
7	0.0000	-0.0000	0.0000	-17.2066	17.2066

2D Graph 4

DEXTROSE (First-order reaction)

50°C / 20 %moisture

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

```

xnear0(q)=max(abs(q))-abs(q)
yatxnear0(q,r)=xatymax(q,xnear0(r))
[Parameters]
a = yatxnear0(y,x) "Auto {{previous: 99.8667}}
b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 2.10922}}
[Equation]
f=a*exp(-b*x)
fit f to y
"fit f to y with weight reciprocal_y
"fit f to y with weight reciprocal_ysquare
[Constraints]
b>0
[Options]
tolerance=0.0001
stepsize=100
iterations=100

```

R = 0.97845723 Rsqr = 0.95737856 Adj Rsqr = 0.94885427

Standard Error of Estimate = 7.8399

	Coefficient		Std. Error	t	P
a	99.8667	7.8390	12.7397	<0.0001	
b	2.1092	0.6285	3.3561	0.0202	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	6903.0535	6903.0535	112.3119	0.0001
Residual	5	307.3163	61.4633		
Total	6	7210.3698	1201.7283		

PRESS = 400202.4543

Durbin-Watson Statistic = 0.4245

Normality Test: Failed (P = 0.0112)

Constant Variance Test: Passed (P = 0.0545)

Power of performed test with alpha = 0.0500: 0.9948

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	99.8667	0.1333	0.0170	1.1699	1.2278
2	12.1171	-2.1559	-0.2750	-1.1575	-1.2100
3	1.4702	8.6146	1.0988	1.1313	1.1731

4	0.0216	6.7143	0.8564	0.8565	0.8293
5	0.0000	7.0723	0.9021	0.9021	0.8818
6	0.0000	8.8752	1.1321	1.1321	1.1741
7	0.0000	7.3872	0.9423	0.9423	0.9293

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	3240.5340 0.9998	84.4919	
2	11.1981 0.9436	-4.9472	
3	0.0384 0.0566	0.2873	
4	0.0000 0.0001	0.0059	
5	0.0000 0.0000	0.0000	
6	0.0000 0.0000	0.0000	
7	0.0000 0.0000	0.0000	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	99.8667	79.7159	120.0176	71.3676	128.3658
2	12.1171	-7.4589	31.6931	-15.9785	40.2127
3	1.4702	-3.3245	6.2649	-19.2453	22.1857
4	0.0216	-0.1220	0.1653	-20.1318	20.1751
5	0.0000	-0.0004	0.0005	-20.1529	20.1530
6	0.0000	-0.0000	0.0000	-20.1530	20.1530
7	0.0000	-0.0000	0.0000	-20.1530	20.1530

2D Graph 5

DEXTROSE (First-order reaction)
50°C
Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y = 1/abs(y)

reciprocal_ysquare = 1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q) = max(abs(q)) - abs(q)

yatxnear0(q,r) = xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 95.1795}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.0531936}}

[Equation]

f = a * exp(-b * x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b > 0

[Options]

tolerance = 0.0001

stepsize = 100

iterations = 100

R = 0.95335268 Rsqr = 0.90888134 Adj Rsqr = 0.89065761

Standard Error of Estimate = 7.2707

	Coefficient		Std. Error	t	P
a	95.1795	4.5093	21.1072	<0.0001	
b	0.0532	0.0090	5.9329	0.0019	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	2636.4745	2636.4745	49.8735	0.0009
Residual	5	264.3162	52.8632		
Total	6	2900.7906	483.4651		

PRESS = 595.5611

Durbin-Watson Statistic = 1.2438

Normality Test: Passed (P = 0.6459)

Constant Variance Test: Passed (P = 0.6019)

Power of performed test with alpha = 0.0500: 0.9620

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	95.1795	4.8205	0.6630	0.8452	0.8165
2	90.2489	1.8105	0.2490	0.2926	0.2640
3	85.5737	3.8791	0.5335	0.5993	0.5564
4	76.9373	-9.3995	-1.2928	-1.4073	-1.6197
5	65.5891	-7.5493	-1.0383	-1.1601	-1.2137
6	55.9148	-0.4379	-0.0602	-0.0716	-0.0641
7	32.8480	8.7874	1.2086	1.6836	2.2881

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.2233	0.3847	0.6456	
2	0.0163	0.2759	0.1630	
3	0.0470	0.2075	0.2847	
4	0.1831	0.1561	-0.6966	
5	0.1671	0.1989	-0.6048	
6	0.0011	0.2923	-0.0412	
7	1.3327	0.4846	2.2189	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	95.1795	83.5879	106.7712	73.1868	117.1723
2	90.2489	80.4312	100.0665	69.1373	111.3605
3	85.5737	77.0603	94.0870	65.0361	106.1112
4	76.9373	69.5534	84.3211	56.8416	97.0329
5	65.5891	57.2538	73.9244	45.1247	86.0535
6	55.9148	45.8099	66.0196	34.6680	77.1615
7	32.8480	19.8368	45.8593	10.0751	55.6210

2D Graph 6

DEXTROSE (Biphasic First-order reaction)

25°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.168671}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.267513}}

c = yatxnear0(y,x)/2 "Auto {{previous: 0.819683}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 4.2155e-012}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.94079403 Rsqr = 0.88509342 Adj Rsqr = 0.77018683

Standard Error of Estimate = 0.0325

	Coefficient		Std. Error	t	P
a	0.1687	0.1299	1.2984	0.2850	
b	0.2675	0.3250	0.8230	0.4708	
c	0.8197	0.1373	5.9708	0.0094	
d	0.0000	0.0090	0.0000	1.0000	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.0243	0.0081	7.7027	0.0638
Residual	3	0.0032	0.0011		
Total	6	0.0275	0.0046		

PRESS = 0.2526

Durbin-Watson Statistic = 3.5677

Normality Test: Passed (P = 0.6858)

Constant Variance Test: Passed (P = 0.4907)

Power of performed test with alpha = 0.0500: 0.9370

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	0.9884	0.0116	0.3588	0.9128	0.8770
2	0.9488	-0.0307	-0.9464	-1.1514	-1.2585
3	0.9185	0.0239	0.7363	0.9658	0.9500
4	0.8775	-0.0144	-0.4442	-0.5757	-0.4984
5	0.8456	0.0281	0.8671	1.1115	1.1833
6	0.8313	-0.0223	-0.6863	-1.1178	-1.1947
7	0.8205	0.0037	0.1146	1.3110	1.6380

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	1.1398	0.8455	2.0514	
2	0.1591	0.3244	-0.8720	
3	0.1680	0.4187	0.8063	
4	0.0563	0.4046	-0.4109	
5	0.1986	0.3914	0.9489	
6	0.5163	0.6231	-1.5360	
7	55.8124	0.9924	18.6680	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	0.9884	0.8934	1.0833	0.8480	1.1287
2	0.9488	0.8899	1.0076	0.8299	1.0676
3	0.9185	0.8516	0.9853	0.7954	1.0415
4	0.8775	0.8118	0.9432	0.7551	0.9999
5	0.8456	0.7810	0.9102	0.7238	0.9674
6	0.8313	0.7498	0.9128	0.6997	0.9629
7	0.8205	0.7176	0.9234	0.6747	0.9663

2D Graph 1

DEXTROSE (Biphasic First-order reaction)

25°C / 60 %RH

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.433192}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.421275}}

c = yatxnear0(y,x)/2 "Auto {{previous: 0.586266}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 1.86902e-010}}

[Equation]

$$f = a \cdot \exp(-b \cdot x) + c \cdot \exp(-d \cdot x)$$

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.89024727 Rsqr = 0.79254021 Adj Rsqr = 0.58508042

Standard Error of Estimate = 0.1194

	Coefficient		Std. Error	t	P
a	0.4332	0.2466	1.7565	0.1773	
b	0.4213	0.4753	0.8864	0.4407	
c	0.5863	0.2447	2.3958	0.0962	
d	0.0000	0.0270	0.0000	1.0000	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.1635	0.0545	3.8202	0.1500
Residual	3	0.0428	0.0143		
Total	6	0.2063	0.0344		

PRESS = 3.5557

Durbin-Watson Statistic = 2.3563

Normality Test: Passed (P = 0.6032)

Constant Variance Test: Passed (P = 0.1815)

Power of performed test with alpha = 0.0500: 0.8123

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0195	-0.0195	-0.1629	-0.5416	-0.4656
2	0.8705	-0.0062	-0.0522	-0.0664	-0.0543
3	0.7728	0.0868	0.7269	0.9972	0.9958
4	0.6666	-0.0390	-0.3264	-0.4020	-0.3375
5	0.6090	-0.1526	-1.2778	-1.6945	-6.6847

6	0.5927	0.0928	0.7769	1.0849	1.1363
7	0.5864	0.0377	0.3153	2.2021	(+inf)

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	0.7373	0.9095	-1.4762
2	0.0007	0.3830	-0.0428
3	0.2192	0.4686	0.9352
4	0.0209	0.3408	-0.2427
5	0.5447	0.4314	-5.8228
6	0.2794	0.4871	1.1073
7	57.9123	0.9795	(+inf)

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0195	0.6569	1.3820	0.4942	1.5448
2	0.8705	0.6353	1.1058	0.4235	1.3176
3	0.7728	0.5126	1.0330	0.3121	1.2335
4	0.6666	0.4447	0.8885	0.2264	1.1068
5	0.6090	0.3593	0.8586	0.1542	1.0638
6	0.5927	0.3274	0.8580	0.1291	1.0562
7	0.5864	0.2101	0.9626	0.0515	1.1212

2D Graph 2

DEXTROSE (Biphasic First-order reaction)
40°C / 75 %RH
Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.912799}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 93.796}}

c = yatxnear0(y,x)/2 "Auto {{previous: 0.0872007}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 0.0233318}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.99961260 Rsqr = 0.99922535 Adj Rsqr = 0.99845071

Standard Error of Estimate = 0.0138

	Coefficient		Std. Error	t	P
a	0.9128	0.0167	54.5026	<0.0001	
b	93.7960	685683.1062	0.0001	0.9999	
c	0.0872	0.0095	9.1641	0.0027	
d	0.0233	0.0139	1.6780	0.1919	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.7350	0.2450	1289.9098	<0.0001
Residual	3	0.0006	0.0002		
Total	6	0.7356	0.1226		

PRESS = 1898897780025.0015

Durbin-Watson Statistic = 2.3452

Normality Test: Passed (P = 0.7511)

Constant Variance Test: Passed (P = 0.6019)

Power of performed test with alpha = 0.0500: 1.0000

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0000	-0.0000	-0.0000	-1.4899	-2.3854
2	0.0852	0.0155	1.1227	1.4238	2.0414
3	0.0832	-0.0029	-0.2086	-0.2497	-0.2060
4	0.0794	-0.0143	-1.0399	-1.1666	-1.2888
5	0.0741	0.0030	0.2142	0.2354	0.1940
6	0.0691	-0.0078	-0.5638	-0.6430	-0.5655
7	0.0547	0.0069	0.5008	0.9322	0.9031

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	2499270892732516.5000	1.0000	-160084122.9027
2	0.3082	0.3782	1.5920
3	0.0067	0.3017	-0.1354
4	0.0880	0.2055	-0.6555
5	0.0029	0.1720	0.0884
6	0.0311	0.2312	-0.3101
7	0.5354	0.7114	1.4177

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0000	0.9561	1.0439	0.9380	1.0620
2	0.0852	0.0582	0.1122	0.0337	0.1367
3	0.0832	0.0591	0.1073	0.0332	0.1333
4	0.0794	0.0595	0.0993	0.0313	0.1276
5	0.0741	0.0559	0.0923	0.0266	0.1215
6	0.0691	0.0480	0.0901	0.0204	0.1177
7	0.0547	0.0177	0.0917	-0.0027	0.1121

2D Graph 3

DEXTROSE (Biphasic First-order reaction)

50°C / 20%moisture

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.908409}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 22045.5}}

c = yatxnear0(y,x)/2 "Auto {{previous: 0.0915914}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 0.0130566}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.99941069 Rsqr = 0.99882173 Adj Rsqr = 0.99764346

Standard Error of Estimate = 0.0168

	Coefficient		Std. Error	t	P
a	0.9084	0.0201	45.0859	<0.0001	
b	22045.4822		837239.9928	0.0263	0.9806
c	0.0916	0.0111	8.2664	0.0037	
d	0.0131	0.0140	0.9319	0.4201	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.7202	0.2401	847.7015	<0.0001
Residual	3	0.0008	0.0003		
Total	6	0.7210	0.1202		

PRESS = 58309481048160.2500

Durbin-Watson Statistic = 1.8654

Normality Test: Passed (P = 0.3935)

Constant Variance Test: Passed (P = 0.8429)

Power of performed test with alpha = 0.0500: 1.0000

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0000	-0.0000	-0.0000	-9.5623	(+inf)
2	0.0904	0.0092	0.5472	0.6820	0.6058
3	0.0892	0.0116	0.6903	0.8213	0.7616
4	0.0869	-0.0196	-1.1630	-1.3071	-1.6266
5	0.0836	-0.0129	-0.7647	-0.8383	-0.7822
6	0.0804	0.0084	0.4975	0.5608	0.4840
7	0.0705	0.0033	0.1979	0.4049	0.3400

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFITS	
1	-51475181805011992.0000		1.0000	(+inf)
2	0.0643	0.3561	0.4505	
3	0.0700	0.2934	0.4908	
4	0.1124	0.2083	-0.8344	
5	0.0355	0.1680	-0.3514	
6	0.0213	0.2131	0.2519	
7	0.1306	0.7611	0.6070	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0000	0.9464	1.0536	0.9243	1.0757
2	0.0904	0.0584	0.1224	0.0280	0.1528
3	0.0892	0.0602	0.1182	0.0283	0.1501
4	0.0869	0.0625	0.1114	0.0281	0.1458
5	0.0836	0.0616	0.1055	0.0257	0.1415
6	0.0804	0.0557	0.1051	0.0214	0.1394
7	0.0705	0.0238	0.1173	-0.0005	0.1416

2D Graph 4

DEXTROSE (Biphasic First-order reaction)

50°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.507061}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.198221}}

c = yatxnear0(y,x)/2 "Auto {{previous: 0.509762}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 0.010782}}

[Equation]

$$f = a \cdot \exp(-b \cdot x) + c \cdot \exp(-d \cdot x)$$

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.98931299 Rsqr = 0.97874020 Adj Rsqr = 0.95748039

Standard Error of Estimate = 0.0453

	Coefficient		Std. Error	t	P
a	0.5071	0.4358	1.1634	0.3288	
b	0.1982	0.1900	1.0433	0.3735	
c	0.5098	0.4515	1.1290	0.3410	
d	0.0108	0.0412	0.2619	0.8104	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.2839	0.0946	46.0371	0.0052
Residual	3	0.0062	0.0021		
Total	6	0.2901	0.0483		

PRESS = 0.6980

Durbin-Watson Statistic = 2.6392

Normality Test: Passed (P = 0.4222)

Constant Variance Test: Passed (P = 0.8429)

Power of performed test with alpha = 0.0500: 0.9995

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0168	-0.0168	-0.3710	-0.8581	-0.8066
2	0.9202	0.0004	0.0091	0.0110	0.0090
3	0.8400	0.0545	1.2028	1.5404	2.7511
4	0.7177	-0.0423	-0.9337	-1.2409	-1.4524
5	0.5993	-0.0189	-0.4171	-0.5293	-0.4538

6	0.5275	0.0273	0.6011	1.0595	1.0935
7	0.4205	-0.0042	-0.0915	-1.2847	-1.5638

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFBETS
1	0.8006	0.8130	-1.6821	
2	0.0000	0.3109	0.0060	
3	0.3798	0.3903	2.2011	
4	0.2951	0.4339	-1.2716	
5	0.0427	0.3788	-0.3544	
6	0.5911	0.6781	1.5870	
7	80.8563	0.9949	-21.8923	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0168	0.8867	1.1469	0.8225	1.2111
2	0.9202	0.8397	1.0006	0.7550	1.0854
3	0.8400	0.7498	0.9301	0.6699	1.0101
4	0.7177	0.6227	0.8128	0.5449	0.8905
5	0.5993	0.5105	0.6881	0.4299	0.7687
6	0.5275	0.4087	0.6463	0.3406	0.7144
7	0.4205	0.2766	0.5644	0.2167	0.6243

2D Graph 5

DiCalcium Phosphate Dihydrate (First-order reaction)

25°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 93.5638}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.00669228}}

[Equation]

f=a*exp(-b*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

[Options]

tolerance=0.0001

stepsize=100

iterations=100

R = 0.22215643 Rsqr = 0.04935348 Adj Rsqr = 0.00000000

Standard Error of Estimate = 17.8133

	Coefficient		Std. Error	t	P
a	93.5638	8.9201	10.4891	<0.0001	
b	0.0067	0.0120	0.5570	0.5977	

Analysis of Variance:

	DF	SS	MS	F	P
Regression1		98.8417	98.8417	0.3115	0.5970
Residual	6	1903.8879	317.3146		
Total	7	2002.7295	286.1042		

PRESS = 3042.9073

Durbin-Watson Statistic = 2.6111

Normality Test: Passed ($P = 0.2724$)

Constant Variance Test: Passed ($P = 0.8849$)

Power of performed test with $\alpha = 0.0500$: 0.0729

The power of the performed test (0.0729) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	93.5638	6.4362	0.3613	0.4174	0.3867
2	92.9397	2.7105	0.1522	0.1711	0.1566
3	92.3198	-13.7590	-0.7724	-0.8509	-0.8283
4	91.7040	29.9978	1.6840	1.8281	2.5074
5	91.0924	-12.5671	-0.7055	-0.7586	-0.7283
6	89.2818	-23.4710	-1.3176	-1.4143	-1.5813
7	87.5071	5.5220	0.3100	0.3448	0.3179
8	81.8426	5.1597	0.2897	0.5839	0.5489

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	0.0292	0.2508	0.2237
2	0.0039	0.2090	0.0805
3	0.0773	0.1760	-0.3828
4	0.2983	0.1515	1.0594
5	0.0450	0.1351	-0.2879
6	0.1522	0.1320	-0.6168
7	0.0141	0.1917	0.1549
8	0.5224	0.7539	0.9608

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	93.5638	71.7371	115.3905	44.8166	142.3109
2	92.9397	73.0151	112.8643	45.0140	140.8654
3	92.3198	74.0355	110.6041	45.0525	139.5871
4	91.7040	74.7404	108.6677	44.9317	138.4763
5	91.0924	75.0696	107.1151	44.6531	137.5317
6	89.2818	73.4427	105.1208	42.9055	135.6580
7	87.5071	68.4207	106.5936	39.9238	135.0905
8	81.8426	43.9956	119.6896	24.1167	139.5685

2D Graph 1

DiCalcium Phosphate Dihydrate (First-order reaction)
25°C/ 60 %RH

NO OUTPUT

DiCalcium Phosphate Dihydrate (First-order reaction)
40°C/ 75 %RH
 Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 88.6789}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.0208095}}

[Equation]

f=a*exp(-b*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

[Options]

tolerance=0.0001

stepsize=100

iterations=100

R = 0.68153155 Rsqr = 0.46448525 Adj Rsqr = 0.37523279

Standard Error of Estimate = 11.3148

	Coefficient		Std. Error	t	P
a	88.6789	5.9606	14.8776	<0.0001	
b	0.0208	0.0098	2.1212	0.0782	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	666.2571	666.2571	5.2042	0.0627
Residual	6	768.1417	128.0236		
Total	7	1434.3988	204.9141		

PRESS = 1338.7404

Durbin-Watson Statistic = 2.1340

Normality Test: Passed (P = 0.5252)

Constant Variance Test: Failed (P = 0.0287)

Power of performed test with alpha = 0.0500: 0.4603

The power of the performed test (0.4603) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	88.6789	11.3211	1.0006	1.1771	1.2254
2	86.8526	-9.9140	-0.8762	-0.9923	-0.9907
3	85.0639	-13.2474	-1.1708	-1.2914	-1.3874
4	83.3120	14.6881	1.2981	1.4075	1.5700
5	81.5963	5.7987	0.5125	0.5503	0.5156
6	76.6581	-9.1053	-0.8047	-0.8689	-0.8484
7	72.0187	-3.7947	-0.3354	-0.3792	-0.3504
8	58.4886	4.4182	0.3905	0.6921	0.6586

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
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1	0.2661	0.2775	0.7594
2	0.1391	0.2203	-0.5266
3	0.1807	0.1781	-0.6458
4	0.1740	0.1494	0.6580
5	0.0232	0.1328	0.2017
6	0.0626	0.1423	-0.3456
7	0.0200	0.2180	-0.1850
8	0.5128	0.6817	0.9638

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	88.6789	74.0939	103.2638	57.3859	119.9718
2	86.8526	73.8590	99.8461	56.2689	117.4362
3	85.0639	73.3804	96.7473	55.0134	115.1143
4	83.3120	72.6105	94.0136	53.6296	112.9945
5	81.5963	71.5077	91.6849	52.1292	111.0633
6	76.6581	66.2136	87.1025	47.0673	106.2488
7	72.0187	59.0921	84.9453	41.4635	102.5740
8	58.4886	35.6301	81.3470	22.5854	94.3917

2D Graph 2

DiCalcium Phosphate Dihydrate (First-order reaction)
50°C/ 20 %moisture
Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 100.426}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.0329605}}

[Equation]

f=a*exp(-b*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

[Options]

tolerance=0.0001

stepsize=100

iterations=100

R = 0.92115552 Rsqr = 0.84852748 Adj Rsqr = 0.82328207

Standard Error of Estimate = 7.1942

	Coefficient		Std. Error	t	P
a	100.4257	3.9556	25.3879	<0.0001	
b	0.0330	0.0065	5.0732	0.0023	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	1739.5926	1739.5926	33.6111	0.0012
Residual	6	310.5385	51.7564		
Total	7	2050.1312	292.8759		

PRESS = 556.3837

Durbin-Watson Statistic = 1.7568

Normality Test: Passed ($P = 0.7068$)

Constant Variance Test: Passed ($P = 0.1196$)

Power of performed test with $\alpha = 0.0500$: 0.9463

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	100.4257	-0.4257	-0.0592	-0.0708	-0.0647
2	97.1696	-4.2745	-0.5942	-0.6769	-0.6429
3	94.0190	-0.9002	-0.1251	-0.1381	-0.1263
4	90.9706	12.1472	1.6885	1.8288	2.5095
5	88.0211	3.3287	0.4627	0.4967	0.4630
6	79.7339	-9.3572	-1.3007	-1.4154	-1.5832
7	72.2270	-4.8509	-0.6743	-0.7750	-0.7458
8	51.9462	4.6423	0.6453	1.0346	1.0419

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.0011	0.3023	-0.0426	
2	0.0682	0.2294	-0.3508	
3	0.0021	0.1790	-0.0590	
4	0.2895	0.1476	1.0442	
5	0.0188	0.1321	0.1806	
6	0.1846	0.1556	-0.6796	
7	0.0964	0.2430	-0.4226	
8	0.8404	0.6110	1.3057	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	100.4257	90.7466	110.1048	80.3367	120.5148
2	97.1696	88.7379	105.6013	77.6509	116.6883
3	94.0190	86.5712	101.4669	74.9048	113.1333
4	90.9706	84.2080	97.7333	72.1128	109.8285
5	88.0211	81.6236	94.4186	69.2911	106.7511
6	79.7339	72.7900	86.6779	60.8103	98.6575
7	72.2270	63.5486	80.9053	52.6005	91.8535
8	51.9462	38.1866	65.7058	29.6031	74.2893

2D Graph 3

DiCalcium Phosphate Dihydrate (First-order reaction)

50°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y = 1/abs(y)

reciprocal_ysquare = 1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q) = max(abs(q)) - abs(q)

yatxnear0(q,r) = xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 82.5879}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.0440508}}

[Equation]

f = a * exp(-b * x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b > 0

[Options]

tolerance = 0.0001

stepsize = 100

Iterations=100

R = 0.79944607 Rsqr = 0.63911401 Adj Rsqr = 0.57896635

Standard Error of Estimate = 12.3137

	Coefficient		Std. Error	t	P
a	82.5879	7.0317	11.7450	<0.0001	
b	0.0441	0.0156	2.8216	0.0303	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	1611.1606	1611.1606	10.6257	0.0173
Residual	6	909.7677	151.6279		
Total	7	2520.9282	360.1326		

PRESS = 1870.5517

Durbin-Watson Statistic = 2.6031

Normality Test: Passed (P = 0.6617)

Constant Variance Test: Passed (P = 0.1196)

Power of performed test with alpha = 0.0500: 0.6891

The power of the performed test (0.6891) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	82.5879	17.4121	1.4140	1.7225	2.2116
2	79.0288	-12.0604	-0.9794	-1.1214	-1.1514
3	75.6231	1.5303	0.1243	0.1372	0.1254
4	72.3641	-14.4028	-1.1697	-1.2658	-1.3497
5	69.2456	9.5619	0.7765	0.8339	0.8096
6	60.6735	-1.3395	-0.1088	-0.1195	-0.1092
7	53.1626	-8.5527	-0.6946	-0.8106	-0.7842
8	34.2213	9.2182	0.7486	1.1062	1.1318

Influence Diagnostics:

Row	Cook's Dist		Leverage	DFFITS
1	0.7179	0.3261	1.5385	
2	0.1954	0.2371	-0.6419	
3	0.0021	0.1792	0.0586	
4	0.1371	0.1462	-0.5584	

5	0.0533	0.1328	0.3169
6	0.0015	0.1708	-0.0495
7	0.1190	0.2658	-0.4718
8	0.7241	0.5420	1.2312

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	82.5879	65.3819	99.7939	47.8906	117.2852
2	79.0288	64.3568	93.7008	45.5158	112.5418
3	75.6231	62.8686	88.3776	42.9041	108.3421
4	72.3641	60.8447	83.8836	40.1066	104.6217
5	69.2456	58.2635	80.2278	37.1760	101.3153
6	60.6735	48.2226	73.1245	28.0717	93.2754
7	53.1626	37.6282	68.6971	19.2632	87.0621
8	34.2213	12.0387	56.4038	-3.1942	71.6368

2D Graph 4

DiCalcium Phosphate Dihydrate (Biphasic First-order reaction)

25°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatznear0(y,x)/2 "Auto {{previous: 0.166203}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.327697}}

c = yatznear0(y,x)/2 "Auto {{previous: 0.837541}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 7.31869e-011}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.34643013 Rsqr = 0.12001384 Adj Rsqr = 0.00000000

Standard Error of Estimate = 0.2099

	Coefficient		Std. Error	t	P
a	0.1662	0.6081	0.2733	0.7981	
b	0.3277	1.9321	0.1696	0.8736	
c	0.8375	0.6331	1.3228	0.2564	
d	0.0000	0.0440	0.0000	1.0000	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.0240	0.0080	0.1818	0.9035
Residual	4	0.1762	0.0441		
Total	7	0.2003	0.0286		

PRESS = 8.8045

Durbin-Watson Statistic = 2.7848

Normality Test: Passed (P = 0.6135)

Constant Variance Test: Passed (P = 0.4979)

Power of performed test with alpha = 0.0500: 0.1247

The power of the performed test (0.1247) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
2	1.0037	-0.0037	-0.0178	-0.0466	-0.0404
3	0.9573	-0.0008	-0.0038	-0.0045	-0.0039
4	0.9238	-0.1382	-0.6586	-0.7957	-0.7511
5	0.8997	0.3173	1.5116	1.8178	3.7747
6	0.8824	-0.0971	-0.4626	-0.5427	-0.4883
7	0.8543	-0.1962	-0.9347	-1.2012	-1.3011
8	0.8438	0.0865	0.4120	0.6309	0.5758
9	0.8378	0.0322	0.1536	1.4559	1.8390

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
2	0.0032	0.8534	-0.0974
3	0.0000	0.2928	-0.0025
4	0.0728	0.3149	-0.5092
5	0.3685	0.3085	2.5211
6	0.0277	0.2735	-0.2996
7	0.2350	0.3945	-1.0502
8	0.1339	0.5736	0.6678
9	47.0686	0.9889	17.3316

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
2	1.0037	0.4654	1.5421	0.2103	1.7971
3	0.9573	0.6420	1.2727	0.2947	1.6199
4	0.9238	0.5968	1.2509	0.2556	1.5921
5	0.8997	0.5760	1.2234	0.2331	1.5664
6	0.8824	0.5776	1.1871	0.2247	1.5400
7	0.8543	0.4883	1.2203	0.1661	1.5425
8	0.8438	0.4024	1.2852	0.1128	1.5749
9	0.8378	0.2582	1.4173	0.0159	1.6597

DiCalcium Phosphate Dihydrate (Biphase First-order reaction)
25°C / 60 %RH

NO OUTPUT

DiCalcium Phosphate Dihydrate (Biphase First-order reaction)
40°C / 75 %RH
Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.333646}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.113982}}

c = yatxnear0(y,x)/2 "Auto {{previous: 0.586469}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 1.50132e-010}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.70718831 Rsqr = 0.50011531 Adj Rsqr = 0.12520180

Standard Error of Estimate = 0.1339

	Coefficient		Std. Error	t	P
a	0.3336	3.9740	0.0840	0.9371	
b	0.1140	1.1744	0.0971	0.9274	
c	0.5865	4.0325	0.1454	0.8914	
d	0.0000	0.2444	0.0000	1.0000	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.0717	0.0239	1.3339	0.3811
Residual	4	0.0717	0.0179		
Total	7	0.1434	0.0205		

PRESS = 22.9003

Durbin-Watson Statistic = 2.1035

Normality Test: Passed (P = 0.7263)

Constant Variance Test: Failed (P = 0.0212)

Power of performed test with alpha = 0.0500: 0.5045

The power of the performed test (0.5045) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	0.9201	0.0799	0.5967	1.2068	1.3106
2	0.8842	-0.1148	-0.8573	-1.0046	-1.0061
3	0.8521	-0.1339	-1.0004	-1.1552	-1.2256
4	0.8235	0.1565	1.1690	1.4051	1.7101
5	0.7980	0.0760	0.5676	0.6926	0.6394
6	0.7367	-0.0612	-0.4569	-0.5619	-0.5070
7	0.6932	-0.0110	-0.0818	-0.1635	-0.1420
8	0.6206	0.0085	0.0632	1.4989	1.9606

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	1.1254	0.7556	2.3042
2	0.0941	0.2716	-0.6144
3	0.1113	0.2501	-0.7078
4	0.2196	0.3079	1.1405
5	0.0587	0.3284	0.4471
6	0.0404	0.3386	-0.3628
7	0.0200	0.7495	-0.2457
8	315.4957	0.9982	46.4673

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	0.9201	0.5970	1.2432	0.4276	1.4127
2	0.8842	0.6904	1.0779	0.4650	1.3034

3	0.8521	0.6662	1.0380	0.4365	1.2677
4	0.8235	0.6172	1.0297	0.3984	1.2486
5	0.7980	0.5849	1.0110	0.3695	1.2264
6	0.7367	0.5204	0.9530	0.3066	1.1668
7	0.6932	0.3714	1.0150	0.2015	1.1849
8	0.6206	0.2492	0.9920	0.0951	1.1461

2D Graph 7

DiCalcium Phosphate Dihydrate (Biphasic First-order reaction)

50°C / 20 %moisture

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.630224}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.0706267}}

c = yatxnear0(y,x)/2 "Auto {{previous: 0.393708}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 6.18862e-010}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y
 "fit f to y with weight reciprocal_ysquare
 [Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.92794829 Rsqr = 0.86108803 Adj Rsqr = 0.75690405

Standard Error of Estimate = 0.0844

	Coefficient		Std. Error	t	P
a	0.6302	9.4315	0.0668	0.9499	
b	0.0706	0.7448	0.0948	0.9290	
c	0.3937	9.4717	0.0416	0.9688	
d	0.0000	0.6242	0.0000	1.0000	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.1765	0.0588	8.2651	0.0345
Residual	4	0.0285	0.0071		
Total	7	0.2050	0.0293		

PRESS = 309.8853

Durbin-Watson Statistic = 1.6439

Normality Test: Passed (P = 0.5092)

Constant Variance Test: Passed (P = 0.9309)

Power of performed test with alpha = 0.0500: 0.9568

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0239	-0.0239	-0.2836	-0.5485	-0.4940
2	0.9810	-0.0520	-0.6163	-0.7225	-0.6711
3	0.9409	-0.0097	-0.1152	-0.1321	-0.1146
4	0.9036	0.1276	1.5120	1.8096	3.6799
5	0.8688	0.0447	0.5294	0.6503	0.5956
6	0.7781	-0.0743	-0.8811	-1.0811	-1.1128
7	0.7047	-0.0310	-0.3668	-0.7862	-0.7404
8	0.5472	0.0187	0.2216	6.7997	(+inf)

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	0.2061 0.7326	-0.8177	
2	0.0489 0.2724	-0.4106	
3	0.0014 0.2389	-0.0642	
4	0.3539 0.3018	2.4196	
5	0.0538 0.3373	0.4249	
6	0.1477 0.3358	-0.7912	
7	0.5552 0.7823	-1.4036	
8	10866.7651	0.9989	(+inf)

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0239	0.8234	1.2245	0.7156	1.3323
2	0.9810	0.8587	1.1032	0.7167	1.2452
3	0.9409	0.8264	1.0554	0.6802	1.2017
4	0.9036	0.7749	1.0323	0.6363	1.1709
5	0.8688	0.7328	1.0049	0.5979	1.1397
6	0.7781	0.6424	0.9139	0.5073	1.0489
7	0.7047	0.4975	0.9119	0.3920	1.0175
8	0.5472	0.3130	0.7813	0.2160	0.8784

2D Graph 8

DiCalcium Phosphate Dihydrate (Biphasic First-order reaction)

50°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.257223}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 356066}}

c = yatxnear0(y,x)/2 "Auto {{previous: 0.742777}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 0.0315182}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.90478094 Rsqr = 0.81862855 Adj Rsqr = 0.68259996

Standard Error of Estimate = 0.1069

	Coefficient		Std. Error	t	P
a	0.2572	0.1277	2.0138	0.1143	
b	356065.7031		5319171.9321	0.0669	0.9498
c	0.7428	0.0699	10.6286	0.0004	
d	0.0315	0.0139	2.2608	0.0866	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.2064	0.0688	6.0181	0.0578
Residual	4	0.0457	0.0114		
Total	7	0.2521	0.0360		

PRESS = 12930026640575624.0000

Durbin-Watson Statistic = 3.1687

Normality Test: Passed (P = 0.7820)

Constant Variance Test: Passed (P = 0.6194)

Power of performed test with alpha = 0.0500: 0.9177

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0000	0.0000	0.0000	11.2065	(+inf)
2	0.7197	-0.0500	-0.4681	-0.5694	-0.5144
3	0.6974	0.0741	0.6934	0.7999	0.7558
4	0.6758	-0.0961	-0.8993	-1.0033	-1.0045
5	0.6548	0.1333	1.2466	1.3639	1.6150
6	0.5957	-0.0024	-0.0222	-0.0243	-0.0210
7	0.5420	-0.0959	-0.8967	-1.0289	-1.0391
8	0.3955	0.0389	0.3642	0.6307	0.5756

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	282793880062915520.0000		1.0000 (+inf)
2	0.0389	0.3242	-0.3563
3	0.0529	0.2486	0.4348
4	0.0616	0.1966	-0.4969
5	0.0917	0.1646	0.7169
6	0.0000	0.1591	-0.0091
7	0.0838	0.2404	-0.5846
8	0.1988	0.6665	0.8137

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0000	0.7032	1.2968	0.5802	1.4198
2	0.7197	0.5507	0.8887	0.3781	1.0613
3	0.6974	0.5494	0.8454	0.3657	1.0291
4	0.6758	0.5441	0.8074	0.3510	1.0005
5	0.6548	0.5344	0.7752	0.3345	0.9751
6	0.5957	0.4773	0.7141	0.2761	0.9153
7	0.5420	0.3964	0.6875	0.2114	0.8726
8	0.3955	0.1531	0.6378	0.0123	0.7787

2D Graph 9

CALCIUM SULFATE (First-order reaction)

25°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y = 1/abs(y)

reciprocal_ysquare = 1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q) = max(abs(q)) - abs(q)

yatxnear0(q,r) = xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 93.7223}}

b = -ln(.5)/(x50(x,y) - min(x)) "Auto {{previous: 0.0127838}}

[Equation]

f = a * exp(-b * x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b > 0

[Options]

tolerance = 0.0001

stepsize = 100

iterations = 100

R = 0.89831165

Rsqr = 0.80696382

Adj Rsqr = 0.77479112

Standard Error of Estimate = 3.6322

	Coefficient		Std. Error	t	P
a	93.7223	1.8595	50.4022	<0.0001	
b	0.0128	0.0027	4.7961	0.0030	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	330.9054	330.9054	25.0823	0.0024
Residual	6	79.1569	13.1928		
Total	7	410.0623	58.5803		

PRESS = 183.7898

Durbin-Watson Statistic = 1.3821

Normality Test: Passed (P = 0.6907)

Constant Variance Test: Passed (P = 0.7053)

Power of performed test with alpha = 0.0500: 0.9053

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	93.7223	6.2777	1.7284	2.0120	3.2203
2	92.5318	-0.6072	-0.1672	-0.1886	-0.1727
3	91.3564	-1.2651	-0.3483	-0.3839	-0.3549
4	90.1960	0.8058	0.2218	0.2407	0.2208
5	89.0502	-5.3088	-1.4616	-1.5706	-1.8683
6	85.6997	-2.0053	-0.5521	-0.5939	-0.5588
7	82.4752	-0.0686	-0.0189	-0.0212	-0.0193
8	72.5779	2.2181	0.6107	1.1624	1.2055

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.7189	0.2621	1.9192	
2	0.0048	0.2139	-0.0901	
3	0.0159	0.1770	-0.1646	
4	0.0051	0.1506	0.0930	
5	0.1908	0.1339	-0.7348	
6	0.0277	0.1358	-0.2215	
7	0.0001	0.2026	-0.0097	
8	1.7722	0.7240	1.9525	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	93.7223	89.1723	98.2723	83.7377	103.7069
2	92.5318	88.4210	96.6426	82.7395	102.3241
3	91.3564	87.6170	95.0958	81.7141	100.9987
4	90.1960	86.7468	93.6451	80.6625	99.7294
5	89.0502	85.7974	92.3030	79.5860	98.5144
6	85.6997	82.4245	88.9749	76.2278	95.1716
7	82.4752	78.4749	86.4756	72.7288	92.2217
8	72.5779	65.0155	80.1402	60.9082	84.2475

2D Graph 5

CALCIUM SULFATE (First-order reaction)

25°C / 60 %RH

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = ~~1~~atxnear0(y,x) "Auto {{previous: 92.3787}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.00639435}}

[Equation]

f=a*exp(-b*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

[Options]

tolerance=0.0001

stepsize=100

iterations=100

R = 0.67039550 Rsqr = 0.44943012 Adj Rsqr = 0.35766848

Standard Error of Estimate = 4.3632

	Coefficient		Std. Error	t	P
a	92.3787	2.1829	42.3187	<0.0001	
b	0.0064	0.0030	2.1532	0.0748	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	93.2440	93.2440	4.8978	0.0688
Residual	6	114.2276	19.0379		
Total	7	207.4715	29.6388		

PRESS = 203.7636

Durbin-Watson Statistic = 1.5918

Normality Test: Passed (P = 0.2602)

Constant Variance Test: Passed (P = 0.0716)

Power of performed test with alpha = 0.0500: 0.4422

The power of the performed test (0.4422) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	92.3787	7.6213	1.7467	2.0173	3.2467
2	91.7898	-3.3997	-0.7792	-0.8759	-0.8562
3	91.2048	-4.3680	-1.0011	-1.1028	-1.1274
4	90.6234	-1.2764	-0.2925	-0.3176	-0.2924
5	90.0458	-2.1758	-0.4987	-0.5362	-0.5017
6	88.3349	0.3927	0.0900	0.0966	0.0882
7	86.6565	4.2343	0.9704	1.0792	1.0974
8	81.2889	-1.0288	-0.2358	-0.4765	-0.4434

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	0.6794 0.2503	1.8760	
2	0.1012 0.2087	-0.4398	
3	0.1298 0.1759	-0.5209	
4	0.0090 0.1515	-0.1235	
5	0.0225 0.1352	-0.1983	
6	0.0007 0.1319	0.0344	
7	0.1378 0.1913	0.5338	
8	0.3500 0.7551	-0.7787	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	92.3787	87.0372	97.7201	80.4406	104.3168
2	91.7898	86.9119	96.6678	80.0518	103.5279
3	91.2048	86.7268	95.6828	79.6272	102.7823
4	90.6234	86.4679	94.7790	79.1667	102.0802
5	90.0458	86.1204	93.9712	78.6706	101.4211
6	88.3349	84.4572	92.2127	76.9760	99.6938
7	86.6565	81.9866	91.3265	75.0034	98.3097
8	81.2889	72.0113	90.5664	67.1446	95.4331

2D Graph 6

CALCIUM SULFATE (First-order reaction)

40°C / 75 %RH

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y = 1/abs(y)

reciprocal_ysquare = 1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q) = max(abs(q)) - abs(q)

yatxnear0(q,r) = xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 89.0732}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.0053762}}

[Equation]

f = a * exp(-b * x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b > 0

[Options]

tolerance = 0.0001

stepsize = 100

iterations = 100

R = 0.47385900 Rsqr = 0.22454235 Adj Rsqr = 0.09529941

Standard Error of Estimate = 5.8884

	Coefficient		Std. Error	t	P
a	89.0732	2.9347	30.3520	<0.0001	
b	0.0054	0.0041	1.3127	0.2373	

Analysis of Variance:

	DF	SS	MS	F	P
Regression 1		60.2402	60.2402	1.7374	0.2356
Residual	6	208.0396	34.6733		
Total	7	268.2798	38.3257		

PRESS = 505.4006

Durbin-Watson Statistic = 1.3856

Normality Test: Passed (P = 0.5123)

Constant Variance Test: Passed (P = 0.4228)

Power of performed test with alpha = 0.0500: 0.2095

The power of the performed test (0.2095) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	89.0732	10.9268	1.8557	2.1404	4.0184
2	88.5956	-1.6784	-0.2850	-0.3203	-0.2949
3	88.1206	-3.8642	-0.6562	-0.7228	-0.6906
4	87.6481	-0.4213	-0.0715	-0.0777	-0.0709
5	87.1781	-1.6130	-0.2739	-0.2946	-0.2709
6	85.7833	-7.5173	-1.2766	-1.3698	-1.5083
7	84.4109	0.8923	0.1515	0.1683	0.1540
8	79.9926	3.2879	0.5584	1.1399	1.1756

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.7570	0.2484	2.3100	
2	0.0135	0.2079	-0.1511	
3	0.0557	0.1757	-0.3188	
4	0.0005	0.1516	-0.0300	
5	0.0068	0.1354	-0.1072	
6	0.1419	0.1314	-0.5866	
7	0.0033	0.1896	0.0745	
8	2.0578	0.7600	2.0923	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	89.0732	81.8923	96.2541	72.9745	105.1718
2	88.5956	82.0263	95.1649	72.7603	104.4309
3	88.1206	82.0808	94.1603	72.4975	103.7436
4	87.6481	82.0374	93.2588	72.1858	103.1104
5	87.1781	81.8762	92.4801	71.8252	102.5311
6	85.7833	80.5607	91.0059	70.4576	101.1091
7	84.4109	78.1378	90.6840	68.6961	100.1256
8	79.9926	67.4313	92.5539	60.8775	99.1078

2D Graph 7

CALCIUM SULFATE (First-order reaction)

50°C / 20 %moisture

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y = 1/abs(y)

reciprocal_ysquare = 1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q) = max(abs(q)) - abs(q)

yatxnear0(q,r) = xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 89.2674}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.0271455}}

[Equation]

f = a * exp(-b * x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b > 0

[Options]

tolerance = 0.0001

stepsize = 100

iterations = 100

R = 0.81445053

Rsqr = 0.66332967

Adj Rsqr = 0.60721794

Standard Error of Estimate = 9.6577

	Coefficient		Std. Error	t	P
a	89.2674	5.2038	17.1544	<0.0001	
b	0.0271	0.0091	2.9908	0.0243	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	1102.6058	1102.6058	11.8216	0.0138
Residual	6	559.6232	93.2705		
Total	7	1662.2290	237.4613		

PRESS = 1019.5279

Durbin-Watson Statistic = 2.1187

Normality Test: Passed (P = 0.2449)

Constant Variance Test: Passed (P = 0.3207)

Power of performed test with alpha = 0.0500: 0.7222

The power of the performed test (0.7222) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	89.2674	10.7326	1.1113	1.3192	1.4292
2	86.8768	-12.1566	-1.2588	-1.4300	-1.6078
3	84.5502	-2.1805	-0.2258	-0.2491	-0.2286
4	82.2860	-6.0993	-0.6315	-0.6844	-0.6507
5	80.0823	-1.6722	-0.1732	-0.1859	-0.1702
6	73.8192	15.2045	1.5743	1.7064	2.1713
7	68.0459	0.4526	0.0469	0.0534	0.0488
8	51.8693	-4.5297	-0.4690	-0.7877	-0.7594

Influence Diagnostics:

Row	Cook's Dist		Leverage	DFFITS
1	0.3560	0.2903	0.9141	
2	0.2971	0.2251	-0.8667	
3	0.0068	0.1787	-0.1066	
4	0.0408	0.1484	-0.2716	
5	0.0026	0.1322	-0.0664	
6	0.2545	0.1488	0.9078	
7	0.0004	0.2310	0.0267	

8 0.5647 0.6454 -1.0245

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	89.2674	76.5343	102.0006	62.4239	116.1110
2	86.8768	75.6639	98.0897	60.7201	113.0336
3	84.5502	74.5615	94.5389	58.8944	110.2060
4	82.2860	73.1815	91.3904	56.9613	107.6106
5	80.0823	71.4892	88.6754	54.9370	105.2276
6	73.8192	64.7037	82.9347	48.4906	99.1478
7	68.0459	56.6884	79.4034	41.8268	94.2649
8	51.8693	32.8841	70.8545	21.5562	82.1824

2D Graph 8

CALCIUM SULFATE (First-order reaction)

50°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 89.2447}}

b = ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.0333771}}

[Equation]
 $f=a*\exp(-b*x)$
 fit f to y
 "fit f to y with weight reciprocal_y
 "fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

[Options]

tolerance=0.0001

stepsize=100

iterations=100

R = 0.88984785 Rsqr = 0.79182919 Adj Rsqr = 0.75713406

Standard Error of Estimate = 7.5621

	Coefficient		Std. Error	t	P
a	89.2447	4.1608	21.4487	<0.0001	
b	0.0334	0.0077	4.3280	0.0049	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	1305.1093	1305.1093	22.8225	0.0031
Residual	6	343.1114	57.1852		
Total	7	1648.2207	235.4601		

PRESS = 991.9402

Durbin-Watson Statistic = 1.2651

Normality Test: Passed (P = 0.5061)

Constant Variance Test: Passed (P = 0.7053)

Power of performed test with alpha = 0.0500: 0.8884

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	89.2447	10.7553	1.4223	1.7033	2.1636
2	86.3151	-2.1575	-0.2853	-0.3250	-0.2994
3	83.4817	0.4318	0.0571	0.0630	0.0575
4	80.7413	1.2402	0.1640	0.1776	0.1626
5	78.0909	-5.5809	-0.7380	-0.7922	-0.7642
6	70.6503	-9.8174	-1.2982	-1.4130	-1.5791
7	63.9186	-3.1203	-0.4126	-0.4744	-0.4414
8	45.7797	9.1538	1.2105	1.9377	2.8914

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.6298	0.3027	1.4257	
2	0.0157	0.2296	-0.1634	
3	0.0004	0.1790	0.0269	
4	0.0027	0.1476	0.0676	
5	0.0477	0.1321	-0.2981	
6	0.1843	0.1559	-0.6785	
7	0.0362	0.2435	-0.2504	
8	2.9330	0.6097	3.6141	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	89.2447	79.0635	99.4259	68.1249	110.3645
2	86.3151	77.4494	95.1809	65.7971	106.8332
3	83.4817	75.6528	91.3107	63.3899	103.5736
4	80.7413	73.6336	87.8491	60.9194	100.5633
5	78.0909	71.3662	84.8156	58.4031	97.7787
6	70.6503	63.3453	77.9553	50.7568	90.5438
7	63.9186	54.7886	73.0487	43.2850	84.5523
8	45.7797	31.3309	60.2285	22.3029	69.2564

2D Graph 9

CALCIUM SULFATE (Biphasic First-order reaction)

25°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.10396}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.831913}}

c = yatxnear0(y,x)/2 "Auto {{previous: 0.893294}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 0.0088255}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.97516606

Rsqr = 0.95094885

Adj Rsqr = 0.91416049

Standard Error of Estimate = 0.0224

	Coefficient		Std. Error	t	P
a	0.1040	0.0334	3.1144	0.0357	
b	0.8319	0.6029	1.3798	0.2398	
c	0.8933	0.0274	32.6200	<0.0001	
d	0.0088	0.0026	3.4236	0.0267	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.0390	0.0130	25.8492	0.0044
Residual	4	0.0020	0.0005		
Total	7	0.0410	0.0059		

PRESS = 0.0191

Durbin-Watson Statistic = 2.7875

Normality Test: Passed (P = 0.2261)

Constant Variance Test: Passed (P = 0.6194)

Power of performed test with alpha = 0.0500: 0.9983

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	0.9973	0.0027	0.1224	0.8138	0.7716
2	0.9307	-0.0114	-0.5104	-0.7925	-0.7475
3	0.8974	0.0036	0.1586	0.1970	0.1715
4	0.8785	0.0315	1.4044	1.6131	2.3632
5	0.8660	-0.0286	-1.2765	-1.5233	-2.0360
6	0.8401	-0.0031	-0.1401	-0.1744	-0.1516
7	0.8179	0.0062	0.2767	0.3262	0.2863
8	0.7488	-0.0008	-0.0353	-0.1181	-0.1025

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	7.1494	0.9774	5.0700	
2	0.2215	0.5852	-0.8879	
3	0.0053	0.3520	0.1264	
4	0.2077	0.2420	1.3353	
5	0.2461	0.2978	-1.3260	
6	0.0042	0.3546	-0.1123	
7	0.0104	0.2804	0.1788	
8	0.0355	0.9107	-0.3271	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	0.9973	0.9357	1.0588	0.9097	1.0848
2	0.9307	0.8831	0.9783	0.8523	1.0091
3	0.8974	0.8604	0.9343	0.8250	0.9697
4	0.8785	0.8479	0.9092	0.8091	0.9479
5	0.8660	0.8321	0.9000	0.7951	0.9370
6	0.8401	0.8030	0.8772	0.7676	0.9125
7	0.8179	0.7849	0.8508	0.7474	0.8883
8	0.7488	0.6893	0.8082	0.6627	0.8348

2D Graph 10

CALCIUM SULFATE (Biphasic First-order reaction)

25°C / 60%RH

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.409319}}

b = ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.00642529}}

c = yatxnear0(y,x)/2 "Auto {{previous: 0.514467}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 0.00636928}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1
iterations=100

R = 0.67039549 Rsqr = 0.44943012 Adj Rsqr = 0.03650270

Standard Error of Estimate = 0.0534

	Coefficient	Std. Error	t	P
a	0.4093	2.7704	0.1477	0.8897
b	0.0064	0.8393	0.0077	0.9943
c	0.5145	2.7704	0.1857	0.8617
d	0.0064	0.6629	0.0096	0.9928

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.0093	0.0031	1.0884	0.4499
Residual	4	0.0114	0.0029		
Total	7	0.0207	0.0030		

PRESS = 0.0223

Durbin-Watson Statistic = 1.5918

Normality Test: Passed (P = 0.2601)

Constant Variance Test: Passed (P = 0.0716)

Power of performed test with alpha = 0.0500: 0.4422

The power of the performed test (0.4422) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	0.9238	0.0762	1.4262	1.6587	2.5712
2	0.9179	-0.0340	-0.6362	-0.7167	-0.6648
3	0.9120	-0.0437	-0.8174	-0.9011	-0.8741
4	0.9062	-0.0128	-0.2389	-0.2599	-0.2270
5	0.9005	-0.0218	-0.4072	-0.4405	-0.3911
6	0.8834	0.0039	0.0735	0.0814	0.0705
7	0.8666	0.0423	0.7923	0.9560	0.9426
8	0.8129	-0.0103	-0.1926	-0.4148	-0.3672

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFITS
1	0.2426	0.2607	1.5270

2	0.0346	0.2121	-0.3449
3	0.0437	0.1771	-0.4055
4	0.0031	0.1551	-0.0973
5	0.0083	0.1456	-0.1614
6	0.0004	0.1846	0.0335
7	0.1041	0.3131	0.6364
8	-0.2425	1.2156	(+inf)

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	0.9238	0.8480	0.9995	0.7572	1.0904
2	0.9179	0.8496	0.9862	0.7546	1.0812
3	0.9120	0.8496	0.9745	0.7511	1.0730
4	0.9062	0.8478	0.9647	0.7468	1.0657
5	0.9005	0.8439	0.9571	0.7417	1.0593
6	0.8834	0.8196	0.9471	0.7219	1.0448
7	0.8666	0.7835	0.9496	0.6966	1.0366
8	0.8129	0.6493	0.9765	0.5920	1.0337

2D Graph 11

CALCIUM SULFATE (Biphasic First-order reaction)

40°C / 75%RH

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.15346}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 1.84686}}

c = yatxnear0(y,x)/2 "Auto {{previous: 0.846437}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 0.00115251}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.91149456 Rsqr = 0.83082234 Adj Rsqr = 0.70393910

Standard Error of Estimate = 0.0337

	Coefficient		Std. Error	t	P
a	0.1535	0.0418	3.6682	0.0214	
b	1.8469	1.6895	1.0931	0.3358	
c	0.8464	0.0253	33.4390	<0.0001	
d	0.0012	0.0029	0.3996	0.7099	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.0223	0.0074	6.5479	0.0505
Residual	4	0.0045	0.0011		
Total	7	0.0268	0.0038		

PRESS = 0.0727

Durbin-Watson Statistic = 2.5967

Normality Test: Passed (P = 0.0534)

Constant Variance Test: Passed (P = 0.1597)

Power of performed test with $\alpha = 0.0500$: 0.9299

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	0.9999	0.0001	0.0031	0.1511	0.1313
2	0.8697	-0.0005	-0.0147	-0.0569	-0.0493
3	0.8483	-0.0057	-0.1705	-0.1954	-0.1700
4	0.8441	0.0282	0.8357	0.9882	0.9843
5	0.8426	0.0130	0.3863	0.4554	0.4050
6	0.8396	-0.0570	-1.6914	-1.8922	-5.0595
7	0.8367	0.0163	0.4837	0.5417	0.4874
8	0.8271	0.0057	0.1679	0.4485	0.3986

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	14.0117	0.9996	6.5020
2	0.0113	0.9330	-0.1840
3	0.0030	0.2387	-0.0952
4	0.0972	0.2848	0.6212
5	0.0202	0.2804	0.2529
6	0.2251	0.2010	-2.5373
7	0.0186	0.2026	0.2457
8	0.3086	0.8599	0.9873

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	0.9999	0.9064	1.0934	0.8676	1.1321
2	0.8697	0.7793	0.9600	0.7396	0.9997
3	0.8483	0.8026	0.8940	0.7442	0.9524
4	0.8441	0.7942	0.8940	0.7381	0.9501
5	0.8426	0.7931	0.8922	0.7368	0.9485
6	0.8396	0.7977	0.8816	0.7371	0.9421
7	0.8367	0.7946	0.8788	0.7342	0.9393
8	0.8271	0.7404	0.9139	0.6996	0.9547

2D Graph 13

CALCIUM SULFATE (Biphasic First-order reaction)

50°C / 20% moisture

Nonlinear Regression

[Variables]

$x = \text{col}(1)$

$y = \text{col}(2)$

$\text{reciprocal_y} = 1/\text{abs}(y)$

$\text{reciprocal_ysquare} = 1/y^2$

'Automatic Initial Parameter Estimate Functions

$\text{xnear0}(q) = \max(\text{abs}(q)) - \text{abs}(q)$

$\text{yatsnear0}(q,r) = \text{xatymax}(q, \text{xnear0}(r))$

[Parameters]

$a = \text{yatsnear0}(y,x)/2$ "Auto {{previous: 0.148785}}

$b = -\ln(.5)/(0.5*(x50(x,y) - \min(x)))$ "Auto {{previous: 46.9667}}

$c = \text{yatsnear0}(y,x)/2$ "Auto {{previous: 0.851214}}

$d = -\ln(.5)/(1.5*(x50(x,y) - \min(x)))$ "Auto {{previous: 0.0225145}}

[Equation]

$f = a * \exp(-b * x) + c * \exp(-d * x)$

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

$b > 0$

$d > 0$

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.87160383 Rsqr = 0.75969324 Adj Rsqr = 0.57946318

Standard Error of Estimate = 0.0999

	Coefficient		Std. Error	t	P
a	0.1488	0.1178	1.2625	0.2754	
b	46.9667	4971732.3006	0.0000	1.0000	
c	0.8512	0.0625	13.6268	0.0002	
d	0.0225	0.0100	2.2500	0.0876	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.1263	0.0421	4.2151	0.0992
Residual	4	0.0399	0.0100		
Total	7	0.1662	0.0237		

PRESS = 0.0000

Durbin-Watson Statistic = 1.8740

Normality Test: Failed (P = 0.0154)

Constant Variance Test: Passed (P = 0.5373)

Power of performed test with alpha = 0.0500: 0.8498

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0000	0.0000	0.0000	(+inf)	(+inf)
2	0.8323	-0.0851	-0.8512	-1.0224	-1.0303
3	0.8137	0.0100	0.0997	0.1146	0.0994
4	0.7956	-0.0338	-0.3378	-0.3769	-0.3323
5	0.7779	0.0062	0.0620	0.0679	0.0588
6	0.7271	0.1631	1.6325	1.7725	3.3137
7	0.6796	0.0054	0.0538	0.0610	0.0528
8	0.5426	-0.0692	-0.6925	-1.2949	-1.4714

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	(+inf)	1.0000	(+inf)	
2	0.1157	0.3069	-0.6856	
3	0.0011	0.2430	0.0563	
4	0.0087	0.1967	-0.1645	
5	0.0002	0.1663	0.0263	
6	0.1404	0.1517	1.4012	

7	0.0003	0.2215	0.0282
8	1.0462	0.7140	-2.3246

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0000	0.7225	1.2775	0.6076	1.3924
2	0.8323	0.6786	0.9860	0.5151	1.1494
3	0.8137	0.6770	0.9505	0.5044	1.1231
4	0.7956	0.6726	0.9187	0.4921	1.0991
5	0.7779	0.6648	0.8910	0.4783	1.0775
6	0.7271	0.6190	0.8352	0.4293	1.0249
7	0.6796	0.5490	0.8102	0.3730	0.9863
8	0.5426	0.3082	0.7770	0.1794	0.9058

2D Graph 14

CALCIUM SULFATE (Biphasic First-order reaction)

50°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.437725}}

$b = -\ln(.5)/(0.5*(x50(x,y)-\min(x)))$ "Auto {{previous: 0.211931}}
 $c = \text{yatznear0}(y,x)/2$ "Auto {{previous: 0.54193}}
 $d = -\ln(.5)/(1.5*(x50(x,y)-\min(x)))$ "Auto {{previous: 2.18758e-011}}

[Equation]

$f = a * \exp(-b * x) + c * \exp(-d * x)$

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

$b > 0$

$d > 0$

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.97882582 Rsqr = 0.95809999 Adj Rsqr = 0.92667497

Standard Error of Estimate = 0.0416

	Coefficient		Std. Error	t	P
a	0.4377	0.2591	1.6896	0.1664	
b	0.2119	0.1610	1.3161	0.2585	
c	0.5419	0.2714	1.9965	0.1166	
d	0.0000	0.0245	0.0000	1.0000	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.1579	0.0526	30.4885	0.0032
Residual	4	0.0069	0.0017		
Total	7	0.1648	0.0235		

PRESS = 0.0772

Durbin-Watson Statistic = 2.4276

Normality Test: Passed (P = 0.4467)

Constant Variance Test: Passed (P = 0.1597)

Power of performed test with alpha = 0.0500: 0.9991

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	0.9797	0.0203	0.4896	1.1091	1.1543
2	0.8961	-0.0545	-1.3112	-1.5403	-2.0912

3	0.8284	0.0107	0.2577	0.3037	0.2661
4	0.7737	0.0461	1.1096	1.3399	1.5630
5	0.7294	-0.0043	-0.1046	-0.1253	-0.1087
6	0.6412	-0.0329	-0.7916	-0.9896	-0.9863
7	0.5945	0.0135	0.3243	0.5626	0.5077
8	0.5482	0.0011	0.0262	0.3785	0.3338

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFBETS
1	1.2704	0.8051	2.3460
2	0.2253	0.2753	-1.2890
3	0.0090	0.2798	0.1659
4	0.2057	0.3143	1.0582
5	0.0017	0.3025	-0.0716
6	0.1378	0.3601	-0.7399
7	0.1589	0.6676	0.7195
8	7.4290	0.9952	4.8078

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	0.9797	0.8761	1.0832	0.8247	1.1347
2	0.8961	0.8355	0.9566	0.7658	1.0263
3	0.8284	0.7674	0.8895	0.6979	0.9589
4	0.7737	0.7090	0.8384	0.6415	0.9060
5	0.7294	0.6660	0.7929	0.5978	0.8611
6	0.6412	0.5720	0.7105	0.5067	0.7758
7	0.5945	0.5002	0.6888	0.4455	0.7435
8	0.5482	0.4332	0.6633	0.3853	0.7112

2D Graph 15

ANNITOL (First-order reaction)

25°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 99.7941}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.00571098}}

[Equation]

f=a*exp(-b*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

[Options]

tolerance=0.0001

stepsize=100

iterations=100

R = 0.81183641

Rsqr = 0.65907835

Adj Rsqr = 0.60225808

Standard Error of Estimate = 2.7275

	Coefficient		Std. Error	t	P
a	99.7941	1.3611	73.3173	<0.0001	
b	0.0057	0.0017	3.3560	0.0153	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	86.2916	86.2916	11.5994	0.0144
Residual	6	44.6361	7.4393		
Total	7	130.9276	18.7039		

PRESS = 191.7129

Durbin-Watson Statistic = 2.0012

Normality Test: Passed (P = 0.4365)

Constant Variance Test: Failed ($P = 0.0212$)

Power of performed test with $\alpha = 0.0500$: 0.7164

The power of the performed test (0.7164) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	99.7941	0.2059	0.0755	0.0871	0.0796
2	99.2258	0.3929	0.1441	0.1619	0.1481
3	98.6608	2.6544	0.9732	1.0720	1.0883
4	98.0989	1.2187	0.4468	0.4851	0.4518
5	97.5403	-1.0840	-0.3974	-0.4274	-0.3962
6	95.8833	-1.1694	-0.4287	-0.4601	-0.4276
7	94.2546	-5.0372	-1.8468	-2.0522	-3.4314
8	89.0225	2.8270	1.0365	2.1085	3.7821

Influence Diagnostics:

Row	Cook's Dist		Leverage	DFFITS
1	0.0013	0.2490	0.0458	
2	0.0034	0.2082	0.0759	
3	0.1225	0.1758	0.5026	
4	0.0210	0.1516	0.1910	
5	0.0143	0.1353	-0.1568	
6	0.0160	0.1316	-0.1664	
7	0.4944	0.1902	-1.6627	
8	6.9770	0.7584	6.7004	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	99.7941	96.4636	103.1247	92.3352	107.2530
2	99.2258	96.1807	102.2709	91.8900	106.5617
3	98.6608	95.8626	101.4589	91.4239	105.8976
4	98.0989	95.5004	100.6974	90.9369	105.2609
5	97.5403	95.0851	99.9954	90.4290	104.6515
6	95.8833	93.4626	98.3041	88.7839	102.9828
7	94.2546	91.3443	97.1649	86.9736	101.5355
8	89.0225	83.2105	94.8346	80.1726	97.8725

2D Graph 10

MANNITOL (First-order reaction)

25°C / 60 %RH

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y = 1/abs(y)

reciprocal_ysquare = 1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q) = max(abs(q)) - abs(q)

yatxnear0(q,r) = xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 96.8219}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.000606115}}

[Equation]

f = a * exp(-b * x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b > 0

[Options]

tolerance = 0.0001

stepsize = 100

iterations = 100

R = 0.10972658 Rsqr = 0.01203992 Adj Rsqr = 0.00000000

Standard Error of Estimate = 3.7103

	Coefficient		Std. Error	t	P
a	96.8219	1.8177	53.2664	<0.0001	
b	0.0006	0.0022	0.2729	0.7941	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	1.0066	1.0066	0.0731	0.7959
Residual	6	82.5959	13.7660		
Total	7	83.6025	11.9432		

PRESS = 266.4003

Durbin-Watson Statistic = 1.1848

Normality Test: Passed (P = 0.6426)

Constant Variance Test: Passed (P = 0.7053)

Power of performed test with alpha = 0.0500: 0.0433

The power of the performed test (0.0433) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	96.8219	3.1781	0.8566	0.9826	0.9792
2	96.7632	0.1484	0.0400	0.0448	0.0409
3	96.7046	4.2603	1.1482	1.2640	1.3470
4	96.6460	0.4894	0.1319	0.1433	0.1310
5	96.5874	-5.0795	-1.3691	-1.4733	-1.6834
6	96.4119	-4.3008	-1.1592	-1.2423	-1.3159
7	96.2368	-1.4615	-0.3939	-0.4356	-0.4040
8	95.6553	2.7657	0.7454	1.5934	1.9152

Influence Diagnostics:

Row	Cook's Dist		Leverage	DFBETS
1	0.1524	0.2400	0.5503	
2	0.0003	0.2040	0.0207	
3	0.1691	0.1747	0.6198	
4	0.0018	0.1522	0.0555	
5	0.1715	0.1365	-0.6692	
6	0.1147	0.1294	-0.5073	

7	0.0211	0.1821	-0.1906
8	4.5310	0.7811	3.6182

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	96.8219	92.3741	101.2696	86.7123	106.9315
2	96.7632	92.6630	100.8634	86.8016	106.7248
3	96.7046	92.9098	100.4993	86.8647	106.5444
4	96.6460	93.1039	100.1881	86.9008	106.3912
5	96.5874	93.2336	99.9412	86.9091	106.2657
6	96.4119	93.1463	99.6776	86.7638	106.0601
7	96.2368	92.3628	100.1108	86.3661	106.1075
8	95.6553	87.6313	103.6792	83.5389	107.7716

2D Graph 11

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MANNITOL (First-order reaction)

40°C / 75 %RH

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 97.8276}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.00540918}}

[Equation]

f=a*exp(-b*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

[Options]

tolerance=0.0001

stepsize=100

iterations=100

R = 0.75578351 Rsqr = 0.57120871 Adj Rsqr = 0.49974349

Standard Error of Estimate = 3.0952

	Coefficient		Std. Error	t	P
a	97.8276	1.5430	63.4009	<0.0001	
b	0.0054	0.0020	2.7573	0.0330	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	76.5729	76.5729	7.9928	0.0301
Residual	6	57.4812	9.5802		
Total	7	134.0541	19.1506		

PRESS = 116.2583

Durbin-Watson Statistic = 2.4989

Normality Test: Passed (P = 0.4876)

Constant Variance Test: Passed (P = 0.4228)

Power of performed test with alpha = 0.0500: 0.5970

The power of the performed test (0.5970) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	97.8276	2.1724	0.7019	0.8096	0.7831
2	97.2999	-0.4606	-0.1488	-0.1672	-0.1530
3	96.7750	0.9734	0.3145	0.3464	0.3194
4	96.2529	-3.6432	-1.1770	-1.2779	-1.3674

5	95.7337	-1.1414	-0.3688	-0.3966	-0.3669
6	94.1927	-2.0666	-0.6677	-0.7164	-0.6839
7	92.6765	5.5524	1.7939	1.9928	3.1284
8	87.7966	-1.3882	-0.4485	-0.9149	-0.9004

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.1084	0.2485	0.4503	
2	0.0037	0.2079	-0.0784	
3	0.0128	0.1757	0.1475	
4	0.1459	0.1516	-0.5781	
5	0.0123	0.1354	-0.1452	
6	0.0388	0.1314	-0.2660	
7	0.4648	0.1897	1.5136	
8	1.3232	0.7597	-1.6009	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	97.8276	94.0520	101.6032	89.3650	106.2902
2	97.2999	93.8463	100.7535	88.9759	105.6238
3	96.7750	93.6001	99.9498	88.5628	104.9872
4	96.2529	93.3038	99.2021	88.1253	104.3805
5	95.7337	92.9469	98.5204	87.6636	103.8038
6	94.1927	91.4471	96.9383	86.1367	102.2487
7	92.6765	89.3780	95.9750	84.4157	100.9373
8	87.7966	81.1954	94.3979	77.7499	97.8434

2D Graph 13

MANNITOL (First-order reaction)
50°C / 20 %moisture
Nonlinear Regression

[Variables]
x = col(1)
y = col(2)
reciprocal_y=1/abs(y)
reciprocal_ysquare=1/y^2
'Automatic Initial Parameter Estimate Functions
xnear0(q)=max(abs(q))-abs(q)
yatxnear0(q,r)=xatymax(q,xnear0(r))
[Parameters]
a = yatxnear0(y,x) "Auto {{previous: 97.1413}}
b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.00848034}}
[Equation]
f=a*exp(-b*x)
fit f to y
"fit f to y with weight reciprocal_y
"fit f to y with weight reciprocal_ysquare
[Constraints]
b>0
[Options]
tolerance=0.0001
stepsize=100
iterations=100

R = 0.82013595 Rsqr = 0.67262297 Adj Rsqr = 0.61806013

Standard Error of Estimate = 3.7282

	Coefficient		Std. Error	t	P
a	97.1413	1.8792	51.6935	<0.0001	
b	0.0085	0.0025	3.4134	0.0143	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	171.3449	171.3449	12.3275	0.0127
Residual	6	83.3965	13.8994		
Total	7	254.7413	36.3916		

PRESS = 139.4052

Durbin-Watson Statistic = 1.2545

Normality Test: Passed (P = 0.5928)

Constant Variance Test: Passed (P = 0.8393)

Power of performed test with alpha = 0.0500: 0.7349

The power of the performed test (0.7349) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	97.1413	2.8587	0.7668	0.8878	0.8696
2	96.3210	4.4557	1.1951	1.3450	1.4691
3	95.5076	-0.3017	-0.0809	-0.0892	-0.0814
4	94.7011	-4.1309	-1.1080	-1.2027	-1.2603
5	93.9014	-2.8757	-0.7714	-0.8292	-0.8045
6	91.5426	-4.2865	-1.1498	-1.2348	-1.3052
7	89.2430	3.2310	0.8666	0.9658	0.9594
8	81.9869	1.0640	0.2854	0.5655	0.5306

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.1342	0.2541	0.5075	
2	0.2411	0.2104	0.7584	
3	0.0009	0.1763	-0.0377	
4	0.1288	0.1512	-0.5319	
5	0.0535	0.1348	-0.3175	
6	0.1170	0.1330	-0.5113	
7	0.1129	0.1949	0.4720	
8	0.4680	0.7453	0.9077	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	97.1413	92.5431	101.7395	86.9254	107.3572
2	96.3210	92.1362	100.5058	86.2844	106.3576
3	95.5076	91.6772	99.3380	85.6135	105.4017
4	94.7011	91.1536	98.2486	84.9131	104.4891
5	93.9014	90.5526	97.2502	84.1836	103.6192
6	91.5426	88.2151	94.8701	81.8321	101.2531
7	89.2430	85.2161	93.2699	79.2712	99.2148
8	81.9869	74.1112	89.8626	69.9350	94.0388

2D Graph 14

MANNITOL (First-order reaction)

50°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 95.6578}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.0224532}}

[Equation]

f=a*exp(-b*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

[Options]

tolerance=0.0001

stepsize=100

iterations=100

R = 0.74699016

Rsqr = 0.55799429

Adj Rsqr = 0.48432668

Standard Error of Estimate = 10.4869

	Coefficient		Std. Error	t	P
a	95.6578	5.5548	17.2206	<0.0001	
b	0.0225	0.0086	2.6065	0.0403	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	833.0008	833.0008	7.5745	0.0332
Residual	6	659.8475	109.9746		
Total	7	1492.8483	213.2640		

PRESS = 1658.5926

Durbin-Watson Statistic = 1.8270

Normality Test: Failed (P = 0.0176)

Constant Variance Test: Passed (P = 0.8393)

Power of performed test with alpha = 0.0500: 0.5794

The power of the performed test (0.5794) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	95.6578	4.3422	0.4141	0.4882	0.4548
2	93.5339	2.1604	0.2060	0.2335	0.2141
3	91.4572	4.6360	0.4421	0.4877	0.4543
4	89.4266	1.6171	0.1542	0.1672	0.1530
5	87.4410	2.0683	0.1972	0.2118	0.1940
6	81.7450	-22.6505	-2.1599	-2.3342	-7.0284
7	76.4201	-1.3635	-0.1300	-0.1473	-0.1347
8	61.0513	9.6455	0.9198	1.6086	1.9472

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.0465	0.2806	0.2840	
2	0.0078	0.2215	0.1142	
3	0.0258	0.1782	0.2116	
4	0.0024	0.1492	0.0640	
5	0.0034	0.1326	0.0759	
6	0.4575	0.1438	-2.8801	
7	0.0031	0.2211	-0.0718	

8 2.6637 0.6731 2.7939

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	95.6578	82.0656	109.2500	66.6198	124.6958
2	93.5339	81.4584	105.6095	65.1741	121.8937
3	91.4572	80.6237	102.2907	63.6036	119.3108
4	89.4266	79.5159	99.3373	61.9187	116.9344
5	87.4410	78.0965	96.7856	60.1321	114.7500
6	81.7450	72.0150	91.4751	54.3018	109.1883
7	76.4201	64.3544	88.4858	48.0645	104.7757
8	61.0513	39.9992	82.1034	27.8601	94.2424

2D Graph 15

MANNITOL (BiPhasic First-order reaction)

25°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.121814}}

b = $\ln(.5)/(0.5*(x50(x,y)-\min(x)))$ "Auto {{previous: 0.133524}}

c = y at x near 0(y,x)/2 "Auto {{previous: 0.895236}}
d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 3.93015e-010}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.89221155 Rsqr = 0.79604144 Adj Rsqr = 0.64307252

Standard Error of Estimate = 0.0258

	Coefficient		Std. Error	t	P
a	0.1218	0.5065	0.2405	0.8218	
b	0.1335	0.5219	0.2558	0.8107	
c	0.8952	0.5172	1.7310	0.1585	
d	0.0000	0.0224	0.0000	1.0000	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.0104	0.0035	5.2039	0.0725
Residual	4	0.0027	0.0007		
Total	7	0.0131	0.0019		

PRESS = 45.5486

Durbin-Watson Statistic = 2.0680

Normality Test: Passed (P = 0.7148)

Constant Variance Test: Passed (P = 0.7941)

Power of performed test with alpha = 0.0500: 0.8932

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0170	-0.0170	-0.6599	-1.3628	-1.6125
2	1.0018	-0.0056	-0.2181	-0.2556	-0.2232
3	0.9885	0.0247	0.9541	1.1057	1.1492

4	0.9768	0.0163	0.6321	0.7610	0.7126
5	0.9666	-0.0021	-0.0805	-0.0979	-0.0849
6	0.9431	0.0041	0.1573	0.1939	0.1687
7	0.9273	-0.0351	-1.3589	-2.6360	(+inf)
8	0.9037	0.0148	0.5739	12.2416	(+inf)

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	1.5161	0.7655	-2.9139
2	0.0061	0.2717	-0.1363
3	0.1049	0.2555	0.6733
4	0.0650	0.3100	0.4776
5	0.0011	0.3239	-0.0588
6	0.0049	0.3414	0.1215
7	4.7995	0.7342	(+inf)
8	17010.3237	0.9978	(+inf)

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0170	0.9543	1.0798	0.9217	1.1124
2	1.0018	0.9644	1.0392	0.9209	1.0827
3	0.9885	0.9522	1.0248	0.9081	1.0689
4	0.9768	0.9369	1.0168	0.8947	1.0589
5	0.9666	0.9258	1.0075	0.8841	1.0492
6	0.9431	0.9012	0.9850	0.8600	1.0262
7	0.9273	0.8658	0.9888	0.8328	1.0218
8	0.9037	0.8320	0.9753	0.8023	1.0051

2D Graph 16

MANNITOL (BiPhasic First-order reaction)

25°C / 60 %RH

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.0547482}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.487789}}

c = yatxnear0(y,x)/2 "Auto {{previous: 0.948324}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 1.26353e-011}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.55878062

Rsqr = 0.31223578

Adj Rsqr = 0.00000000

Standard Error of Estimate = 0.0379

	Coefficient		Std. Error	t	P
a	0.0547	0.0695	0.7882	0.4746	
b	0.4878	1.2185	0.4003	0.7094	
c	0.9483	0.0662	14.3342	0.0001	
d	0.0000	0.0048	0.0000	1.0000	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.0026	0.0009	0.6053	0.6455
Residual	4	0.0057	0.0014		
Total	7	0.0084	0.0012		

PRESS = 1.6045

Durbin-Watson Statistic = 1.5317

Normality Test: Passed (P = 0.7430)

Constant Variance Test: Passed (P = 0.7053)

Power of performed test with alpha = 0.0500: 0.2915

The power of the performed test (0.2915) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0031	-0.0031	-0.0810	-0.2768	-0.2420
2	0.9819	-0.0128	-0.3382	-0.4224	-0.3742
3	0.9690	0.0407	1.0731	1.3410	1.5653
4	0.9610	0.0104	0.2732	0.3218	0.2824
5	0.9561	-0.0410	-1.0821	-1.2497	-1.3862
6	0.9501	-0.0290	-0.7652	-1.0112	-1.0151
7	0.9487	-0.0010	-0.0261	-0.0348	-0.0301
8	0.9483	0.0359	0.9464	5.6130	(+inf)

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.2043	0.9143	-0.7905	
2	0.0250	0.3589	-0.2800	
3	0.2525	0.3596	1.1731	
4	0.0100	0.2792	0.1757	
5	0.1303	0.2503	-0.8009	
6	0.1908	0.4273	-0.8769	
7	0.0002	0.4387	-0.0266	
8	269.1707	0.9716	(+inf)	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0031	0.9024	1.1037	0.8574	1.1487
2	0.9819	0.9189	1.0450	0.8592	1.1047
3	0.9690	0.9058	1.0321	0.8462	1.0917
4	0.9610	0.9054	1.0166	0.8419	1.0801
5	0.9561	0.9034	1.0088	0.8384	1.0738
6	0.9501	0.8813	1.0189	0.8244	1.0759
7	0.9487	0.8790	1.0185	0.8225	1.0750
8	0.9483	0.8446	1.0521	0.8005	1.0961

2D Graph 17

MANNITOL (BiPhasic First-order reaction)

40°C / 75 %RH

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.323691}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.00543067}}

c = yatxnear0(y,x)/2 "Auto {{previous: 0.654584}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 0.00539824}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

Stepsize=0.1
Iterations=100

R = 0.75578349 Rsqr = 0.57120869 Adj Rsqr = 0.24961521

Standard Error of Estimate = 0.0379

	Coefficient	Std. Error	t	P
a	0.3237	753959.2906	0.0000	1.0000
b	0.0054	215.4067	0.0000	1.0000
c	0.6546	753959.2906	0.0000	1.0000
d	0.0054	106.9689	0.0001	1.0000

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.0077	0.0026	1.7762	0.2906
Residual	4	0.0057	0.0014		
Total	7	0.0134	0.0019		

PRESS = 0.0572

Durbin-Watson Statistic = 2.4989

Normality Test: Passed (P = 0.4877)

Constant Variance Test: Passed (P = 0.4228)

Power of performed test with alpha = 0.0500: 0.5970

The power of the performed test (0.5970) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	0.9783	0.0217	0.5731	0.7641	0.7161
2	0.9730	-0.0046	-0.1215	-0.1376	-0.1194
3	0.9677	0.0097	0.2568	0.2581	0.2254
4	0.9625	-0.0364	-0.9610	-1.0366	-1.0497
5	0.9573	-0.0114	-0.3011	-0.3345	-0.2938
6	0.9419	-0.0207	-0.5452	-0.6668	-0.6125
7	0.9268	0.0555	1.4647	1.8386	4.0451
8	0.8780	-0.0139	-0.3663	-1.4318	-1.7760

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	0.1135	0.4375	0.6315

2	0.0013	0.2200	-0.0634
3	0.0002	0.0103	0.0230
4	0.0439	0.1404	-0.4242
5	0.0066	0.1898	-0.1422
6	0.0551	0.3316	-0.4314
7	0.4865	0.3654	3.0693
8	7.3193	0.9346	-6.7114

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	0.9783	0.9087	1.0479	0.8521	1.1045
2	0.9730	0.9236	1.0224	0.8567	1.0892
3	0.9677	0.9571	0.9784	0.8620	1.0735
4	0.9625	0.9231	1.0020	0.8501	1.0749
5	0.9573	0.9115	1.0032	0.8425	1.0721
6	0.9419	0.8813	1.0025	0.8205	1.0634
7	0.9268	0.8631	0.9904	0.8038	1.0497
8	0.8780	0.7762	0.9797	0.7316	1.0244

2D Graph 18

MANNITOL (BiPhasic First-order reaction)

50°C / 20 %moisture

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

```

xnear0(q)=max(abs(q))-abs(q)
yatxnear0(q,r)=xatymax(q,xnear0(r))
[Parameters]
a = yatxnear0(y,x)/2 "Auto {{previous: 0.093512}}
b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.488568}}
c = yatxnear0(y,x)/2 "Auto {{previous: 0.921222}}
d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 0.00453147}}

```

```

[Equation]
f=a*exp(-b*x)+c*exp(-d*x)
fit f to y
"fit f to y with weight reciprocal_y
"fit f to y with weight reciprocal_ysquare

```

```

[Constraints]
b>0
d>0
[Options]
tolerance=1e-6
stepsize=0.1
iterations=100

```

R = 0.90606896 Rsqr = 0.82096096 Adj Rsqr = 0.68668169

Standard Error of Estimate = 0.0338

	Coefficient		Std. Error	t	P
a	0.0935	0.0647	1.4451	0.2219	
b	0.4886	0.6485	0.7534	0.4932	
c	0.9212	0.0623	14.7985	0.0001	
d	0.0045	0.0047	0.9683	0.3877	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.0209	0.0070	6.1138	0.0564
Residual	4	0.0046	0.0011		
Total	7	0.0255	0.0036		

PRESS = 0.1631

Durbin-Watson Statistic = 2.6143

Normality Test: Passed (P = 0.1765)

Constant Variance Test: Passed (P = 0.8393)

Power of performed test with alpha = 0.0500: 0.9201

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0147	-0.0147	-0.4363	-1.4949	-1.9487
2	0.9744	0.0333	0.9873	1.2347	1.3593
3	0.9481	0.0040	0.1170	0.1463	0.1270
4	0.9304	-0.0247	-0.7307	-0.8608	-0.8259
5	0.9179	-0.0077	-0.2270	-0.2625	-0.2293
6	0.8955	-0.0230	-0.6799	-0.9002	-0.8730
7	0.8811	0.0436	1.2919	1.7180	2.9059
8	0.8414	-0.0109	-0.3228	-1.8256	-3.8717

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	5.9980 0.9148	-6.3851	
2	0.2149 0.3606	1.0208	
3	0.0030 0.3602	0.0953	
4	0.0718 0.2794	-0.5143	
5	0.0058 0.2523	-0.1332	
6	0.1525 0.4296	-0.7576	
7	0.5668 0.4345	2.5470	
8	25.8207 0.9687	-21.5526	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0147	0.9251	1.1044	0.8850	1.1445
2	0.9744	0.9181	1.0307	0.8651	1.0838
3	0.9481	0.8918	1.0044	0.8388	1.0574
4	0.9304	0.8808	0.9799	0.8243	1.0364
5	0.9179	0.8708	0.9650	0.8130	1.0228
6	0.8955	0.8341	0.9570	0.7834	1.0076
7	0.8811	0.8193	0.9429	0.7688	0.9934
8	0.8414	0.7491	0.9337	0.7099	0.9730

2D Graph 19

MANNITOL (BiPhasic First-order reaction)

50°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.367652}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.200456}}

c = yatxnear0(y,x)/2 "Auto {{previous: 0.668022}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 3.69366e-010}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.85831342

Rsqr = 0.73670192

Adj Rsqr = 0.53922836

Standard Error of Estimate = 0.0991

	Coefficient		Std. Error	t	P
a	0.3677	0.6963	0.5280	0.6254	
b	0.2005	0.4714	0.4253	0.6925	
c	0.6680	0.7276	0.9181	0.4105	
d	0.0000	0.0520	0.0000	1.0000	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.1100	0.0367	3.7306	0.1179
Residual	4	0.0393	0.0098		
Total	7	0.1493	0.0213		

PRESS = 55.3088

Durbin-Watson Statistic = 2.4780

Normality Test: Failed (P = 0.0438)

Constant Variance Test: Passed (P = 0.8849)

Power of performed test with alpha = 0.0500: 0.8206

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0357	-0.0357	-0.3599	-0.8043	-0.7608
2	0.9689	-0.0119	-0.1205	-0.1415	-0.1229
3	0.9142	0.0467	0.4710	0.5537	0.4990
4	0.8695	0.0409	0.4128	0.4984	0.4457
5	0.8329	0.0622	0.6272	0.7527	0.7036
6	0.7584	-0.1674	-1.6892	-2.1065	(+inf)
7	0.7176	0.0330	0.3330	0.5861	0.5309
8	0.6747	0.0323	0.3256	4.9393	(+inf)

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	0.6462	0.7998	-1.5207
2	0.0019	0.2744	-0.0756
3	0.0293	0.2763	0.3083
4	0.0284	0.3141	0.3016
5	0.0623	0.3056	0.4668
6	0.6158	0.3570	(+inf)
7	0.1801	0.6771	0.7687
8	1397.8128	0.9957	(+inf)

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0357	0.7895	1.2818	0.6664	1.4049
2	0.9689	0.8247	1.1131	0.6582	1.2796
3	0.9142	0.7696	1.0589	0.6033	1.2252
4	0.8695	0.7153	1.0238	0.5540	1.1850
5	0.8329	0.6808	0.9851	0.5184	1.1474
6	0.7584	0.5940	0.9228	0.4378	1.0790
7	0.7176	0.4911	0.9440	0.3611	1.0740
8	0.6747	0.4001	0.9493	0.2859	1.0635

2D Graph 20

LACTOSE HYDROUS (First-order reaction)

25°C

Nonlinear Regression

[Variables]

$x = \text{col}(1)$

$y = \text{col}(2)$

$\text{reciprocal_y} = 1/\text{abs}(y)$

$\text{reciprocal_ysquare} = 1/y^2$

'Automatic Initial Parameter Estimate Functions

$\text{xnear0}(q) = \max(\text{abs}(q)) - \text{abs}(q)$

$\text{yatsnear0}(q,r) = \text{xatymax}(q, \text{xnear0}(r))$

[Parameters]

$a = \text{yatsnear0}(y,x)$ "Auto {{previous: 98.7481}}

$b = -\ln(.5)/(\text{x50}(x,y) - \min(x))$ "Auto {{previous: 0.0143815}}

[Equation]

$f = a * \exp(-b * x)$

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

$b > 0$

[Options]

tolerance=0.0001

stepsize=100

iterations=100

R = 0.85870060 Rsqr = 0.73736673 Adj Rsqr = 0.69359452

Standard Error of Estimate = 5.1813

	Coefficient		Std. Error	t	P
a	98.7481	2.6669	37.0274	<0.0001	
b	0.0144	0.0037	3.9008	0.0080	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	452.2358	452.2358	16.8455	0.0063
Residual	6	161.0761	26.8460		
Total	7	613.3120	87.6160		

PRESS = 296.5965

Durbin-Watson Statistic = 2.2560

Normality Test: Passed (P = 0.6251)

Constant Variance Test: Passed (P = 0.2327)

Power of performed test with alpha = 0.0500: 0.8215

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	98.7481	1.2519	0.2416	0.2818	0.2590
2	97.3382	-0.4754	-0.0918	-0.1036	-0.0946
3	95.9483	2.5909	0.5000	0.5513	0.5165
4	94.5783	7.3974	1.4277	1.5489	1.8252
5	93.2279	-8.9998	-1.7370	-1.8662	-2.6301
6	89.2911	-2.6968	-0.5205	-0.5602	-0.5254
7	85.5206	-1.6393	-0.3164	-0.3549	-0.3275
8	74.0650	2.6254	0.5067	0.9514	0.9425

Influence Diagnostics:

Row	Cook's Dist		Leverage	DFFITS
1	0.0143	0.2649	0.1555	
2	0.0015	0.2151	-0.0495	
3	0.0327	0.1773	0.2397	
4	0.2123	0.1504	0.7679	
5	0.2687	0.1337	-1.0332	
6	0.0249	0.1369	-0.2092	
7	0.0163	0.2054	-0.1665	
8	1.1429	0.7163	1.4977	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	98.7481	92.2225	105.2738	84.4891	113.0072
2	97.3382	91.4577	103.2186	83.3626	111.3137
3	95.9483	90.6106	101.2860	82.1923	109.7043
4	94.5783	89.6617	99.4949	80.9801	108.1765
5	93.2279	88.5922	97.8636	79.7287	106.7270
6	89.2911	84.6004	93.9818	75.7730	102.8093
7	85.5206	79.7749	91.2663	71.6012	99.4401
8	74.0650	63.3346	84.7954	57.4554	90.6746

2D Graph 21

LACTOSE HYDROUS (First-order reaction)

25°C / 60 %RH

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 93.9494}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.0138593}}

[Equation]

f=a*exp(-b*x)

fit f to y
 "fit f to y with weight reciprocal_y
 "fit f to y with weight reciprocal_ysquare
 [Constraints]
 b>0
 [Options]
 Tolerance=0.0001
 stepsize=100
 iterations=100

R = 0.71848585 Rsqr = 0.51622191 Adj Rsqr = 0.43559223

Standard Error of Estimate = 7.5634

	Coefficient		Std. Error	t	P
a	93.9494	3.8869	24.1708	<0.0001	
b	0.0139	0.0056	2.4659	0.0487	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	366.2527	366.2527	6.4024	0.0447
Residual	6	343.2342	57.2057		
Total	7	709.4869	101.3553		

PRESS = 1328.3486

Durbin-Watson Statistic = 2.2353

Normality Test: Passed (P = 0.6128)

Constant Variance Test: Passed (P = 0.4228)

Power of performed test with alpha = 0.0500: 0.5249

The power of the performed test (0.5249) is below the desired power of 0.8000.
 You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	93.9494	6.0506	0.8000	0.9325	0.9206
2	92.6563	1.2016	0.1589	0.1793	0.1641
3	91.3810	5.7777	0.7639	0.8421	0.8187
4	90.1233	-3.3359	-0.4411	-0.4785	-0.4454
5	88.8828	-5.2034	-0.6880	-0.7392	-0.7078
6	85.2630	-0.0518	-0.0069	-0.0074	-0.0067
7	81.7907	-12.7367	-1.6840	-1.8881	-2.7057

8 71.2055 8.4481 1.1170 2.1055 3.7615

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.1560	0.2641	0.5515	
2	0.0044	0.2148	0.0858	
3	0.0764	0.1772	0.3799	
4	0.0203	0.1505	-0.1874	
5	0.0422	0.1338	-0.2781	
6	0.0000	0.1366	-0.0027	
7	0.4584	0.2046	-1.3721	
8	5.6602	0.7186	6.0107	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	93.9494	84.4385	103.4603	73.1415	114.7573
2	92.6563	84.0793	101.2334	72.2583	113.0543
3	91.3810	83.5908	99.1713	71.3012	111.4609
4	90.1233	82.9448	97.3018	70.2728	109.9738
5	88.8828	82.1140	95.6517	69.1768	108.5889
6	85.2630	78.4238	92.1022	65.5327	104.9934
7	81.7907	73.4201	90.1612	61.4786	102.1027
8	71.2055	55.5171	86.8938	46.9436	95.4673

2D Graph 22

LACTOSE HYDROUS (First-order reaction)
40°C / 75 %RH
Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y = 1/abs(y)

reciprocal_ysquare = 1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q) = max(abs(q)) - abs(q)

yatxnear0(q,r) = xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 86.3848}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.0167549}}

[Equation]

f = a * exp(-b * x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b > 0

[Options]

tolerance = 0.0001

stepsize = 100

iterations = 100

R = 0.76339834 Rsqr = 0.58277703 Adj Rsqr = 0.51323986

Standard Error of Estimate = 7.1704

	Coefficient		Std. Error	t	P
a	86.3848	3.7229	23.2036	<0.0001	
b	0.0168	0.0060	2.7776	0.0321	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	430.8915	430.8915	8.3808	0.0275
Residual	6	308.4848	51.4141		
Total	7	739.3762	105.6252		

PRESS = 837.5987

Durbin-Watson Statistic = 1.2449

Normality Test: Passed (P = 0.1090)

Constant Variance Test: Passed (P = 0.8849)

Power of performed test with $\alpha = 0.0500$: 0.6125

The power of the performed test (0.6125) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	86.3848	13.6152	1.8988	2.2217	4.8166
2	84.9495	-1.5968	-0.2227	-0.2517	-0.2310
3	83.5380	-4.2025	-0.5861	-0.6463	-0.6116
4	82.1500	-4.4393	-0.6191	-0.6715	-0.6375
5	80.7851	-0.1747	-0.0244	-0.0262	-0.0239
6	76.8248	-6.9567	-0.9702	-1.0455	-1.0553
7	73.0587	-1.7090	-0.2383	-0.2682	-0.2463
8	61.7883	5.6432	0.7870	1.4457	1.6349

Influence Diagnostics:

Row	Cook's	Dist	Leverage	DFFITS
1	0.9109	0.2696	2.9261	
2	0.0088	0.2171	-0.1216	
3	0.0451	0.1776	-0.2842	
4	0.0398	0.1500	-0.2678	
5	0.0001	0.1333	-0.0094	
6	0.0881	0.1388	-0.4236	
7	0.0096	0.2100	-0.1270	
8	2.4815	0.7037	2.5193	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	86.3848	77.2752	95.4944	66.6156	106.1540
2	84.9495	76.7752	93.1238	65.5935	104.3055
3	83.5380	76.1443	90.9318	64.4985	102.5776
4	82.1500	75.3543	88.9458	63.3347	100.9654
5	80.7851	74.3789	87.1913	62.1069	99.4633
6	76.8248	70.2888	83.3608	58.1017	95.5479
7	73.0587	65.0184	81.0990	53.7589	92.3585
8	61.7883	47.0707	76.5060	38.8876	84.6891

2D Graph 23

LACTOSE HYDROUS (First-order reaction)

50°C / 20 %moisture

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 80.5994}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.0464301}}

[Equation]

f=a*exp(-b*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

[Options]

tolerance=0.0001

stepsize=100

iterations=100

R = 0.83098356 Rsqr = 0.69053367 Adj Rsqr = 0.63895595

Standard Error of Estimate = 11.2615

	Coefficient		Std. Error	t	P
a	80.5994	6.4810	12.4362	<0.0001	
b	0.0464	0.0151	3.0818	0.0216	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	1697.9183	1697.9183	13.3882	0.0106
Residual	6	760.9311	126.8218		
Total	7	2458.8494	351.2642		

PRESS = 1454.5475

Durbin-Watson Statistic = 1.2147

Normality Test: Passed (P = 0.6231)

Constant Variance Test: Passed (P = 0.2897)

Power of performed test with alpha = 0.0500: 0.7592

The power of the performed test (0.7592) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	80.5994	19.4006	1.7227	2.1065	3.7683
2	76.9427	-1.7701	-0.1572	-0.1801	-0.1649
3	73.4519	-12.3742	-1.0988	-1.2128	-1.2743
4	70.1195	-10.3242	-0.9168	-0.9920	-0.9904
5	66.9383	-1.0011	-0.0889	-0.0955	-0.0872
6	58.2347	-4.9448	-0.4391	-0.4832	-0.4499
7	50.6627	9.4856	0.8423	0.9861	0.9834
8	31.8453	2.5046	0.2224	0.3235	0.2979

Influence Diagnostics:

Row	Cook's Dist		Leverage	DFFITS
1	1.0988	0.3312	2.6518	
2	0.0051	0.2386	-0.0923	
3	0.1605	0.1792	-0.5953	
4	0.0841	0.1459	-0.4094	
5	0.0007	0.1332	-0.0342	
6	0.0246	0.1742	-0.2067	

7	0.1802	0.2704	0.5987
8	0.0583	0.5272	0.3146

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	80.5994	64.7409	96.4579	48.8060	112.3928
2	76.9427	63.4814	90.4040	46.2746	107.6109
3	73.4519	61.7883	85.1155	43.5292	103.3746
4	70.1195	59.5932	80.6458	40.6215	99.6175
5	66.9383	56.8811	76.9955	37.6044	96.2722
6	58.2347	46.7322	69.7371	28.3744	88.0950
7	50.6627	36.3334	64.9921	19.6037	81.7217
8	31.8453	11.8369	51.8537	-2.2086	65.8992

2D Graph 24

LACTOSE HYDROUS (First-order reaction)

50°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 95.4743}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.0541414}}

[Equation]

f=a*exp(-b*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

[Options]

tolerance=0.0001

stepsize=100

iterations=100

R = 0.9670 Rsqr = 0.9352 Adj Rsqr = 0.9244

Standard Error of Estimate = 5.7663

	Coefficient		Std. Error	t	P
a	95.4743	3.4038	28.0493	<0.0001	
b	0.0541	0.0072	7.5649	0.0003	

Analysis of Variance:

	DF	SS	MS	F	P
Regression 1		2878.0139	2878.0139	86.5556	<0.0001
Residual 6	6	199.5029	33.2505		
Total 7	7	3077.5168	439.6453		

PRESS = 362.9285

Durbin-Watson Statistic = 0.9032

Normality Test: Passed (P = 0.4645)

Constant Variance Test: Passed (P = 0.6620)

Power of performed test with alpha = 0.0500: 0.9955

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	95.4743	4.5257	0.7848	0.9723	0.9670
2	90.4427	6.3309	1.0979	1.2623	1.3446
3	85.6762	-0.6116	-0.1061	-0.1171	-0.1070
4	81.1609	-5.7712	-1.0009	-1.0826	-1.1017
5	76.8836	-8.9200	-1.5469	-1.6632	-2.0681
6	65.3572	-0.2466	-0.0428	-0.0474	-0.0433
7	55.5589	1.0417	0.1807	0.2136	0.1958

8 32.3311 4.9544 0.8592 1.1889 1.2413

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFBETS
1	0.2528	0.3484	0.7072	
2	0.2564	0.2435	0.7627	
3	0.0015	0.1789	-0.0499	
4	0.0996	0.1453	-0.4542	
5	0.2157	0.1349	-0.8167	
6	0.0003	0.1864	-0.0207	
7	0.0091	0.2849	0.1236	
8	0.6463	0.4777	1.1871	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	95.4743	87.1455	103.8032	79.0898	111.8589
2	90.4427	83.4808	97.4046	74.7089	106.1764
3	85.6762	79.7083	91.6441	70.3563	100.9961
4	81.1609	75.7826	86.5391	66.0609	96.2608
5	76.8836	71.7011	82.0660	61.8522	91.9149
6	65.3572	59.2656	71.4489	49.9887	80.7257
7	55.5589	48.0275	63.0903	39.5650	71.5528
8	32.3311	22.5792	42.0830	15.1794	49.4828

2D Graph 26

LACTOSE HYDROUS (BiPhasic First-order reaction)

25°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.288768}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.0957106}}

c = yatxnear0(y,x)/2 "Auto {{previous: 0.722904}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 7.91853e-011}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.88330073 Rsqr = 0.78022017 Adj Rsqr = 0.61538530

Standard Error of Estimate = 0.0581

	Coefficient		Std. Error	t	P
a	0.2888	2.7836	0.1037	0.9224	
b	0.0957	0.7316	0.1308	0.9022	
c	0.7229	2.8101	0.2573	0.8097	
d	0.0000	0.1240	0.0000	1.0000	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.0479	0.0160	4.7333	0.0836
Residual	4	0.0135	0.0034		
Total	7	0.0613	0.0088		

PRESS = 1.0056

Durbin-Watson Statistic = 2.6048

Normality Test: Passed (P = 0.2530)

Constant Variance Test: Passed (P = 0.1597)

Power of performed test with alpha = 0.0500: 0.8748

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0117	-0.0117	-0.2011	-0.3989	-0.3525
2	0.9853	-0.0167	-0.2875	-0.3369	-0.2960
3	0.9614	0.0240	0.4139	0.4764	0.4248
4	0.9396	0.0802	1.3808	1.6570	2.5624
5	0.9198	-0.0775	-1.3357	-1.6348	-2.4578
6	0.8707	-0.0047	-0.0815	-0.1000	-0.0867
7	0.8338	0.0050	0.0865	0.1780	0.1547
8	0.7655	0.0014	0.0244	0.6448	0.5899

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	0.1168	0.7459	-0.6040
2	0.0106	0.2718	-0.1808
3	0.0184	0.2452	0.2421
4	0.3020	0.3055	1.6996
5	0.3328	0.3324	-1.7345
6	0.0013	0.3369	-0.0618
7	0.0256	0.7637	0.2782
8	72.2003	0.9986	15.5475

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0117	0.8725	1.1509	0.7987	1.2246
2	0.9853	0.9013	1.0693	0.8036	1.1671
3	0.9614	0.8816	1.0412	0.7815	1.1412
4	0.9396	0.8505	1.0287	0.7554	1.1238
5	0.9198	0.8269	1.0127	0.7338	1.1059
6	0.8707	0.7771	0.9642	0.6843	1.0570
7	0.8338	0.6929	0.9746	0.6197	1.0478
8	0.7655	0.6044	0.9265	0.5376	0.9933

2D Graph 26

LACTOSE HYDROUS (BiPhasic First-order reaction)

25°C / 60 %RH

Nonlinear Regression

[Variables]

$x = \text{col}(1)$

$y = \text{col}(2)$

$\text{reciprocal_y} = 1/\text{abs}(y)$

$\text{reciprocal_ysquare} = 1/y^2$

'Automatic Initial Parameter Estimate Functions

$\text{xnear0}(q) = \max(\text{abs}(q)) - \text{abs}(q)$

$\text{yatsnear0}(q,r) = \text{xatymax}(q, \text{xnear0}(r))$

[Parameters]

$a = \text{yatsnear0}(y,x)/2$ "Auto {{previous: 0.255963}}

$b = -\ln(.5)/(0.5*(x50(x,y) - \min(x)))$ "Auto {{previous: 0.234103}}

$c = \text{yatsnear0}(y,x)/2$ "Auto {{previous: 0.753922}}

$d = -\ln(.5)/(1.5*(x50(x,y) - \min(x)))$ "Auto {{previous: 2.42093e-010}}

[Equation]

$f = a * \exp(-b * x) + c * \exp(-d * x)$

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

$b > 0$

$d > 0$

[Options]

tolerance=1e-6

stepsize=0.1
iterations=100

R = 0.88085926 Rsqr = 0.77591304 Adj Rsqr = 0.60784783

Standard Error of Estimate = 0.0630

	Coefficient		Std. Error	t	P
a	0.2560	0.3159	0.8102	0.4633	
b	0.2341	0.3986	0.5873	0.5886	
c	0.7539	0.3323	2.2689	0.0858	
d	0.0000	0.0225	0.0000	1.0000	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.0551	0.0184	4.6167	0.0868
Residual	4	0.0159	0.0040		
Total	7	0.0709	0.0101		

PRESS = 47.4032

Durbin-Watson Statistic = 3.1523

Normality Test: Passed (P = 0.2432)

Constant Variance Test: Passed (P = 0.1388)

Power of performed test with alpha = 0.0500: 0.8697

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0099	-0.0099	-0.1568	-0.3655	-0.3219
2	0.9565	-0.0179	-0.2836	-0.3337	-0.2931
3	0.9142	0.0574	0.9105	1.0784	1.1090
4	0.8807	-0.0129	-0.2040	-0.2464	-0.2150
5	0.8543	-0.0175	-0.2772	-0.3303	-0.2901
6	0.8036	0.0485	0.7689	0.9665	0.9560
7	0.7786	-0.0880	-1.3960	-2.3521	(+inf)
8	0.7563	0.0402	0.6383	8.3457	(+inf)

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.1480	0.8159	-0.6777	
2	0.0107	0.2777	-0.1817	
3	0.1172	0.2872	0.7040	
4	0.0070	0.3142	-0.1455	

5	0.0115	0.2960	-0.1881
6	0.1354	0.3671	0.7281
7	2.5431	0.6477	(+inf)
8	2959.1823	0.9942	(+inf)

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0099	0.8518	1.1680	0.7740	1.2458
2	0.9565	0.8642	1.0487	0.7586	1.1543
3	0.9142	0.8204	1.0080	0.7156	1.1128
4	0.8807	0.7826	0.9788	0.6801	1.0814
5	0.8543	0.7590	0.9495	0.6550	1.0535
6	0.8036	0.6976	0.9097	0.5990	1.0083
7	0.7786	0.6377	0.9194	0.5539	1.0032
8	0.7563	0.5818	0.9308	0.5091	1.0035

2D Graph 27

LACTOSE HYDROUS (BiPhasic First-order reaction)

40°C / 75 %RH

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

$a = \text{yatkxnear0}(y,x)/2$ "Auto {{previous: 0.206606}}
 $b = -\ln(.5)/(0.5*(x50(x,y)-\min(x)))$ "Auto {{previous: 1.381}}
 $c = \text{yatkxnear0}(y,x)/2$ "Auto {{previous: 0.792722}}
 $d = -\ln(.5)/(1.5*(x50(x,y)-\min(x)))$ "Auto {{previous: 0.00910134}}

[Equation]

$f = a * \exp(-b * x) + c * \exp(-d * x)$

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

$b > 0$

$d > 0$

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.97241729 Rsqr = 0.94559538 Adj Rsqr = 0.90479192

Standard Error of Estimate = 0.0317

	Coefficient		Std. Error	t	P
a	0.2066	0.0416	4.9619	0.0077	
b	1.3810	0.7609	1.8149	0.1437	
c	0.7927	0.0283	28.0546	<0.0001	
d	0.0091	0.0035	2.6313	0.0581	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.0699	0.0233	23.1744	0.0054
Residual	4	0.0040	0.0010		
Total	7	0.0739	0.0106		

PRESS = 0.0895

Durbin-Watson Statistic = 2.6635

Normality Test: Passed (P = 0.3537)

Constant Variance Test: Failed (P = 0.0287)

Power of performed test with alpha = 0.0500: 0.9976

Regression Diagnostics:

Row	Predicted Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
-----	--------------------	-----------	------------	-----------------

1	0.9993	0.0007	0.0212	0.4248	0.3765
2	0.8375	-0.0039	-0.1242	-0.3117	-0.2733
3	0.7915	0.0019	0.0593	0.0692	0.0599
4	0.7747	0.0025	0.0775	0.0911	0.0789
5	0.7652	0.0409	1.2897	1.5544	2.1391
6	0.7438	-0.0451	-1.4230	-1.6253	-2.4155
7	0.7238	-0.0103	-0.3236	-0.3666	-0.3230
8	0.6608	0.0135	0.4263	1.1210	1.1722

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	18.0943	0.9975	7.5397
2	0.1286	0.8412	-0.6288
3	0.0004	0.2636	0.0359
4	0.0008	0.2764	0.0488
5	0.2734	0.3116	1.4391
6	0.2012	0.2335	-1.3332
7	0.0095	0.2208	-0.1719
8	1.8580	0.8554	2.8508

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	0.9993	0.9114	1.0873	0.8749	1.1238
2	0.8375	0.7567	0.9182	0.7180	0.9569
3	0.7915	0.7463	0.8367	0.6925	0.8904
4	0.7747	0.7284	0.8209	0.6752	0.8741
5	0.7652	0.7161	0.8144	0.6644	0.8660
6	0.7438	0.7013	0.7864	0.6460	0.8416
7	0.7238	0.6824	0.7651	0.6265	0.8210
8	0.6608	0.5794	0.7422	0.5409	0.7807

2D Graph 28

LACTOSE HYDROUS (BiPhasic First-order reaction)

50°C / 20 %moisture

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.315899}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 1.61813}}

c = yatxnear0(y,x)/2 "Auto {{previous: 0.686347}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 0.0286976}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.96591365 Rsqr = 0.93298917 Adj Rsqr = 0.88273105

Standard Error of Estimate = 0.0642

	Coefficient		Std. Error	t	P
a	0.3159	0.0878	3.5962	0.0228	
b	1.6181	1.3311	1.2157	0.2910	
c	0.6863	0.0616	11.1483	0.0004	
d	0.0287	0.0105	2.7444	0.0517	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.2294	0.0765	18.5639	0.0082
Residual	4	0.0165	0.0041		

Total 7 0.2459 0.0351

PRESS = 6.4486

Durbin-Watson Statistic = 2.9292

Normality Test: Passed (P = 0.2917)

Constant Variance Test: Passed (P = 0.1196)

Power of performed test with alpha = 0.0500: 0.9950

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0022	-0.0022	-0.0350	-1.1714	-1.2516
2	0.7296	0.0222	0.3453	1.1166	1.1656
3	0.6605	-0.0497	-0.7744	-0.9027	-0.8761
4	0.6322	-0.0342	-0.5335	-0.6421	-0.5872
5	0.6124	0.0470	0.7318	0.8779	0.8462
6	0.5614	-0.0285	-0.4447	-0.4996	-0.4468
7	0.5151	0.0864	1.3456	1.5426	2.0991
8	0.3866	-0.0431	-0.6718	-1.4034	-1.7058

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	384.0570	0.9991	-41.8789	
2	2.9478	0.9044	3.5845	
3	0.0731	0.2640	-0.5247	
4	0.0463	0.3098	-0.3933	
5	0.0846	0.3051	0.5607	
6	0.0163	0.2076	-0.2287	
7	0.1870	0.2392	1.1770	
8	1.6562	0.7708	-3.1285	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0022	0.8241	1.1804	0.7503	1.2542
2	0.7296	0.5601	0.8990	0.4837	0.9755
3	0.6605	0.5689	0.7520	0.4601	0.8608
4	0.6322	0.5330	0.7314	0.4283	0.8361
5	0.6124	0.5140	0.7108	0.4088	0.8160
6	0.5614	0.4803	0.6426	0.3656	0.7573
7	0.5151	0.4280	0.6023	0.3168	0.7135
8	0.3866	0.2302	0.5431	0.1495	0.6237

2D Graph 29

LACTOSE HYDROUS (BiPhasic First-order reaction)

50°C

Nonlinear Regression

[Variables]

$x = \text{col}(1)$

$y = \text{col}(2)$

$\text{reciprocal_y} = 1/\text{abs}(y)$

$\text{reciprocal_ysquare} = 1/y^2$

'Automatic Initial Parameter Estimate Functions

$\text{xnear0}(q) = \max(\text{abs}(q)) - \text{abs}(q)$

$\text{yatznear0}(q,r) = \text{xatymax}(q, \text{xnear0}(r))$

[Parameters]

$a = \text{yatznear0}(y,x)/2$ "Auto {{previous: 0.241149}}

$b = -\ln(.5)/(0.5*(x50(x,y) - \min(x)))$ "Auto {{previous: 0.398833}}

$c = \text{yatznear0}(y,x)/2$ "Auto {{previous: 0.783533}}

$d = -\ln(.5)/(1.5*(x50(x,y) - \min(x)))$ "Auto {{previous: 0.0358982}}

[Equation]

$f = a * \exp(-b * x) + c * \exp(-d * x)$

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

$b > 0$

$d > 0$

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.98839567 Rsqr = 0.97692600 Adj Rsqr = 0.95962050

Standard Error of Estimate = 0.0421

	Coefficient		Std. Error	t	P
a	0.2411	0.1465	1.6466	0.1750	
b	0.3988	0.3448	1.1566	0.3118	
c	0.7835	0.1501	5.2208	0.0064	
d	0.0359	0.0134	2.6705	0.0558	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.3007	0.1002	56.4518	0.0010
Residual	4	0.0071	0.0018		
Total	7	0.3078	0.0440		

PRESS = 0.1257

Durbin-Watson Statistic = 2.1388

Normality Test: Passed (P = 0.6862)

Constant Variance Test: Passed (P = 0.2327)

Power of performed test with alpha = 0.0500: 0.9999

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0247	-0.0247	-0.5858	-1.7854	-3.4311
2	0.9177	0.0500	1.1866	1.4512	1.8264
3	0.8379	0.0128	0.3035	0.3755	0.3311
4	0.7764	-0.0225	-0.5346	-0.6368	-0.5818
5	0.7276	-0.0480	-1.1394	-1.3299	-1.5420
6	0.6242	0.0269	0.6382	0.8509	0.8143
7	0.5517	0.0143	0.3401	0.4669	0.4158
8	0.3822	-0.0094	-0.2229	-1.1328	-1.1903

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	6.6056	0.8923	-9.8783	
2	0.2610	0.3314	1.2860	
3	0.0187	0.3470	0.2414	
4	0.0424	0.2951	-0.3764	
5	0.1602	0.2659	-0.9281	

6	0.1408	0.4375	0.7181
7	0.0482	0.4695	0.3911
8	7.9642	0.9613	-5.9310

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0247	0.9142	1.1352	0.8638	1.1856
2	0.9177	0.8504	0.9851	0.7828	1.0527
3	0.8379	0.7690	0.9068	0.7021	0.9736
4	0.7764	0.7129	0.8400	0.6433	0.9096
5	0.7276	0.6673	0.7880	0.5960	0.8593
6	0.6242	0.5468	0.7016	0.4840	0.7645
7	0.5517	0.4715	0.6318	0.4099	0.6935
8	0.3822	0.2676	0.4969	0.2184	0.5461

2D Graph 30

LACTOSE ANHYDROUS (First-order reaction)

25°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yattxnear0(y,x) "Auto {{previous: 94.4043}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.0216548}}

[Equation]

f=a*exp(-b*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

[Options]

tolerance=0.0001

stepsize=100

iterations=100

R = 0.94008231 Rsqr = 0.88375476 Adj Rsqr = 0.86438055

Standard Error of Estimate = 4.1858

	Coefficient		Std. Error	t	P
a	94.4043	2.2117	42.6834	<0.0001	
b	0.0217	0.0034	6.2780	0.0008	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	799.2164	799.2164	45.6150	0.0005
Residual	6	105.1254	17.5209		
Total	7	904.3419	129.1917		

PRESS = 355.8653

Durbin-Watson Statistic = 1.2262

Normality Test: Passed (P = 0.7290)

Constant Variance Test: Passed (P = 0.4228)

Power of performed test with alpha = 0.0500: 0.9731

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	94.4043	5.5957	1.3368	1.5746	1.8765
2	92.3820	-0.5158	-0.1232	-0.1396	-0.1277
3	90.4030	1.2047	0.2878	0.3175	0.2923
4	88.4664	-0.1959	-0.0468	-0.0507	-0.0463
5	86.5712	-2.1704	-0.5185	-0.5568	-0.5219

6	81.1260	-6.1835	-1.4773	-1.5959	-1.9203
7	76.0232	-2.3627	-0.5645	-0.6390	-0.6042
8	61.2211	4.8507	1.1588	2.0389	3.3582

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.4802	0.2792	1.1678	
2	0.0028	0.2209	-0.0680	
3	0.0109	0.1782	0.1361	
4	0.0002	0.1493	-0.0194	
5	0.0237	0.1327	-0.2041	
6	0.2127	0.1431	-0.7848	
7	0.0575	0.2197	-0.3206	
8	4.3554	0.6769	4.8612	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	94.4043	88.9924	99.8162	82.8201	105.9885
2	92.3820	87.5679	97.1960	81.0648	103.6992
3	90.4030	86.0797	94.7263	79.2856	101.5203
4	88.4664	84.5091	92.4236	77.4862	99.4465
5	86.5712	82.8404	90.3021	75.6706	97.4719
6	81.1260	77.2513	85.0007	70.1753	92.0767
7	76.0232	71.2225	80.8240	64.7117	87.3348
8	61.2211	52.7941	69.6481	47.9576	74.4845

2D Graph 16

LACTOSE ANHYDROUS (First-order reaction)
25°C / 60 %RH
Nonlinear Regression

[Variables]
 $x = \text{col}(1)$
 $y = \text{col}(2)$
 $\text{reciprocal_y} = 1/\text{abs}(y)$
 $\text{reciprocal_ysquare} = 1/y^2$
'Automatic Initial Parameter Estimate Functions
 $\text{xnear0}(q) = \max(\text{abs}(q)) - \text{abs}(q)$
 $\text{yatznear0}(q,r) = \text{xatymax}(q, \text{xnear0}(r))$
[Parameters]
 $a = \text{yatznear0}(y,x)$ "Auto {{previous: 92.638}}}
 $b = -\ln(.5)/(\text{x50}(x,y) - \min(x))$ "Auto {{previous: 0.0258057}}}
[Equation]
 $f = a * \exp(-b * x)$
fit f to y
"fit f to y with weight reciprocal_y
"fit f to y with weight reciprocal_ysquare
[Constraints]
 $b > 0$
[Options]
tolerance=0.0001
stepsize=100
iterations=100

R = 0.91391868 Rsqr = 0.83524736 Adj Rsqr = 0.80778859

Standard Error of Estimate = 5.7165

	Coefficient		Std. Error	t	P
a	92.6380	3.0648	30.2261	<0.0001	
b	0.0258	0.0051	5.0800	0.0023	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	994.0183	994.0183	30.4182	0.0015
Residual	6	196.0702	32.6784		
Total	7	1190.0885	170.0126		

PRESS = 598.5089

Durbin-Watson Statistic = 1.3746

Normality Test: Passed ($P = 0.5403$)

Constant Variance Test: Passed ($P = 0.8393$)

Power of performed test with $\alpha = 0.0500$: 0.9342

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	92.6380	7.3620	1.2878	1.5256	1.7802
2	90.2780	-0.6919	-0.1210	-0.1374	-0.1256
3	87.9782	3.3852	0.5922	0.6534	0.6189
4	85.7369	-3.2285	-0.5648	-0.6121	-0.5771
5	83.5527	-2.4399	-0.4268	-0.4582	-0.4258
6	77.3283	-8.1106	-1.4188	-1.5364	-1.8009
7	71.5676	-2.3579	-0.4125	-0.4695	-0.4367
8	55.2897	6.4973	1.1366	1.9313	2.8661

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	0.4695	0.2874	1.1307
2	0.0027	0.2241	-0.0675
3	0.0464	0.1786	0.2885
4	0.0327	0.1486	-0.2411
5	0.0160	0.1323	-0.1663
6	0.2038	0.1473	-0.7484
7	0.0326	0.2281	-0.2373
8	3.5194	0.6536	3.9374

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	92.6380	85.1386	100.1374	76.7667	108.5094
2	90.2780	83.6568	96.8993	74.8023	105.7538
3	87.9782	82.0675	93.8888	72.7929	103.1634
4	85.7369	80.3439	91.1298	70.7455	100.7283
5	83.5527	78.4646	88.6408	68.6682	98.4371
6	77.3283	71.9606	82.6960	62.3460	92.3106
7	71.5676	64.8876	78.2477	56.0667	87.0686
8	55.2897	43.9808	66.5985	37.3022	73.2771

2D Graph 17

LACTOSE ANHYDROUS (First-order reaction)

40°C / 75 %RH

Nonlinear Regression

[Variables]

$x = \text{col}(1)$

$y = \text{col}(2)$

$\text{reciprocal_y} = 1/\text{abs}(y)$

$\text{reciprocal_ysquare} = 1/y^2$

'Automatic Initial Parameter Estimate Functions

$\text{xnear0}(q) = \max(\text{abs}(q)) - \text{abs}(q)$

$\text{yatznear0}(q,r) = \text{xatymax}(q, \text{xnear0}(r))$

[Parameters]

$a = \text{yatznear0}(y,x)$ "Auto {{previous: 84.4599}}

$b = -\ln(.5)/(\text{x50}(x,y) - \min(x))$ "Auto {{previous: 0.0265749}}

[Equation]

$f = a * \exp(-b * x)$

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

$b > 0$

[Options]

tolerance=0.0001

stepsize=100

iterations=100

R = 0.82549980 Rsqr = 0.68144992 Adj Rsqr = 0.62835824

Standard Error of Estimate = 8.1258

	Coefficient		Std. Error	t	P
a	84.4599	4.3666	19.3422	<0.0001	
b	0.0266	0.0080	3.3241	0.0159	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	847.4934	847.4934	12.8353	0.0116
Residual	6	396.1686	66.0281		
Total	7	1243.6620	177.6660		

PRESS = 1026.1832

Durbin-Watson Statistic = 1.2736

Normality Test: Failed (P = 0.0173)

Constant Variance Test: Passed (P = 0.8849)

Power of performed test with alpha = 0.0500: 0.7469

The power of the performed test (0.7469) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	84.4599	15.5401	1.9124	2.2677	5.4759
2	82.2449	-3.9467	-0.4857	-0.5516	-0.5168
3	80.0881	-3.5230	-0.4336	-0.4784	-0.4453
4	77.9878	-1.2118	-0.1491	-0.1616	-0.1479
5	75.9425	-5.1174	-0.6298	-0.6761	-0.6421
6	70.1231	-6.9791	-0.8589	-0.9305	-0.9182
7	64.7496	-1.2352	-0.1520	-0.1732	-0.1585
8	49.6391	6.9853	0.8597	1.4528	1.6472

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	1.0440	0.2888	3.4892	
2	0.0441	0.2246	-0.2781	
3	0.0249	0.1786	-0.2076	
4	0.0023	0.1485	-0.0618	
5	0.0348	0.1323	-0.2507	
6	0.0752	0.1480	-0.3826	

7	0.0045	0.2294	-0.0865
8	1.9586	0.6499	2.2440

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	84.4599	73.7752	95.1446	61.8878	107.0320
2	82.2449	72.8227	91.6672	60.2423	104.2475
3	80.0881	71.6851	88.4910	58.5023	101.6738
4	77.9878	70.3245	85.6511	56.6790	99.2965
5	75.9425	68.7112	83.1739	54.7853	97.0998
6	70.1231	62.4749	77.7713	48.8198	91.4264
7	64.7496	55.2262	74.2730	42.7035	86.7957
8	49.6391	33.6107	65.6675	24.1000	75.1782

2D Graph 18

LACTOSE ANHYDROUS (First-order reaction)

50°C / 20 %moisture

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 75.7282}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.0314973}}

[Equation]

f=a*exp(-b*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

[Options]

tolerance=0.0001

stepsize=100

iterations=100

R = 0.65684694 Rsqr = 0.43144790 Adj Rsqr = 0.33668922

Standard Error of Estimate = 13.3489

	Coefficient		Std. Error	t	P
a	75.7282	7.2998	10.3740	<0.0001	
b	0.0315	0.0157	2.0110	0.0910	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	811.3322	811.3322	4.5531	0.0768
Residual	6	1069.1549	178.1925		
Total	7	1880.4871	268.6410		

PRESS = 2492.0799

Durbin-Watson Statistic = 0.8292

Normality Test: Passed (P = 0.4898)

Constant Variance Test: Passed (P = 0.2897)

Power of performed test with alpha = 0.0500: 0.4209

The power of the performed test (0.4209) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	75.7282	24.2718	1.8183	2.1718	4.2865
2	73.3801	2.5987	0.1947	0.2216	0.2031
3	71.1049	-9.5538	-0.7157	-0.7898	-0.7617
4	68.9002	-10.8148	-0.8102	-0.8776	-0.8581

5	66.7638	-12.0277	-0.9010	-0.9672	-0.9610
6	60.7441	-1.8355	-0.1375	-0.1495	-0.1367
7	55.2671	-2.1270	-0.1593	-0.1827	-0.1673
8	40.3344	10.6059	0.7945	1.2896	1.3847

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	1.0061 0.2990	2.7998	
2	0.0073 0.2283	0.1105	
3	0.0680 0.1789	-0.3556	
4	0.0668 0.1478	-0.3574	
5	0.0712 0.1321	-0.3749	
6	0.0020 0.1537	-0.0582	
7	0.0053 0.2398	-0.0939	
8	1.3591 0.6204	1.7703	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	75.7282	57.8662	93.5902	38.4997	112.9566
2	73.3801	57.7740	88.9863	37.1799	109.5804
3	71.1049	57.2882	84.9215	35.6393	106.5704
4	68.9002	56.3425	81.4578	33.9059	103.8945
5	66.7638	54.8930	78.6347	32.0101	101.5176
6	60.7441	47.9395	73.5486	25.6604	95.8277
7	55.2671	39.2731	71.2611	18.8979	91.6362
8	40.3344	14.6064	66.0624	-1.2449	81.9136

2D Graph 19

LACTOSE ANHYDROUS (First-order reaction)

50°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 87.9779}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.057004}}

[Equation]

f=a*exp(-b*x)

fit f to y

'fit f to y with weight reciprocal_y

'fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

[Options]

tolerance=0.0001

stepsize=100

iterations=100

R = 0.82517354 Rsqr = 0.68091137 Adj Rsqr = 0.62772993

Standard Error of Estimate = 13.3636

	Coefficient		Std. Error	t	P
a	87.9779	7.9557	11.0585	<0.0001	
b	0.0570	0.0186	3.0672	0.0220	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	2286.5353	2286.5353	12.8036	0.0117
Residual	6	1071.5160	178.5860		
Total	7	3358.0512	479.7216		

PRESS = 2205.0436

Durbin-Watson Statistic = 1.9746

Normality Test: Passed (P = 0.4475)

Constant Variance Test: Passed (P = 0.6194)

Power of performed test with $\alpha = 0.0500$: 0.7462

The power of the performed test (0.7462) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	87.9779	12.0221	0.8996	1.1196	1.1492
2	83.1031	5.7646	0.4314	0.4964	0.4628
3	78.4984	-12.3779	-0.9262	-1.0221	-1.0267
4	74.1488	-9.1772	-0.6867	-0.7427	-0.7115
5	70.0402	11.7537	0.8795	0.9460	0.9363
6	59.0307	-15.8898	-1.1890	-1.3217	-1.4331
7	49.7518	-4.2700	-0.3195	-0.3791	-0.3503
8	28.1347	15.7308	1.1771	1.6030	1.9353

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.3441	0.3544	0.8514	
2	0.0400	0.2450	0.2636	
3	0.1137	0.1788	-0.4790	
4	0.0468	0.1452	-0.2932	
5	0.0702	0.1357	0.3709	
6	0.2058	0.1907	-0.6957	
7	0.0293	0.2895	-0.2236	
8	1.0978	0.4608	1.7889	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	87.9779	68.5111	107.4447	49.9225	126.0334
2	83.1031	66.9174	99.2888	46.6169	119.5892
3	78.4984	64.6728	92.3239	42.9961	114.0006
4	74.1488	61.6906	86.6070	39.1564	109.1412
5	70.0402	57.9956	82.0848	35.1929	104.8875
6	59.0307	44.7509	73.3105	23.3491	94.7123
7	49.7518	32.1569	67.3466	12.6190	86.8845
8	28.1347	5.9385	50.3310	-11.3866	67.6561

2D Graph 20

LACTOSE ANHYDROUS (BiPhasic First-order reaction)

25°C

Nonlinear Regression

[Variables]

$x = \text{col}(1)$

$y = \text{col}(2)$

$\text{reciprocal_y} = 1/\text{abs}(y)$

$\text{reciprocal_ysquare} = 1/y^2$

'Automatic Initial Parameter Estimate Functions

$\text{xnear0}(q) = \max(\text{abs}(q)) - \text{abs}(q)$

$\text{yatznear0}(q,r) = \text{xatymax}(q, \text{xnear0}(r))$

[Parameters]

$a = \text{yatznear0}(y,x)/2$ "Auto {{previous: 0.350559}}

$b = -\ln(.5)/(0.5*(x50(x,y) - \min(x)))$ "Auto {{previous: 0.141782}}

$c = \text{yatznear0}(y,x)/2$ "Auto {{previous: 0.640758}}

$d = -\ln(.5)/(1.5*(x50(x,y) - \min(x)))$ "Auto {{previous: 1.12922e-012}}

[Equation]

$f = a * \exp(-b * x) + c * \exp(-d * x)$

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

$b > 0$

$d > 0$

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.99082047 Rsqr = 0.98172520 Adj Rsqr = 0.96801910

Standard Error of Estimate = 0.0203

	Coefficient		Std. Error	t	P
a	0.3506	0.3387	1.0349	0.3592	
b	0.1418	0.1340	1.0577	0.3498	
c	0.6408	0.3469	1.8469	0.1385	
d	0.0000	0.0218	0.0000	1.0000	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.0888	0.0296	71.6269	0.0006
Residual	4	0.0017	0.0004		
Total	7	0.0904	0.0129		

PRESS = 0.0704

Durbin-Watson Statistic = 2.7497

Normality Test: Passed (P = 0.1963)

Constant Variance Test: Passed (P = 0.3207)

Power of performed test with alpha = 0.0500: 1.0000

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	0.9913	0.0087	0.4272	0.8906	0.8614
2	0.9450	-0.0263	-1.2946	-1.5171	-2.0163
3	0.9048	0.0113	0.5567	0.6462	0.5914
4	0.8699	0.0128	0.6318	0.7610	0.7126
5	0.8396	0.0044	0.2180	0.2647	0.2312
6	0.7707	-0.0213	-1.0464	-1.2909	-1.4636
7	0.7257	0.0109	0.5376	1.0295	1.0400
8	0.6613	-0.0006	-0.0301	-0.6127	-0.5574

Influence Diagnostics:

Row	Cook's Dist		Leverage	DFFITS
1	0.6637	0.7699	1.5759	
2	0.2147	0.2718	-1.2318	
3	0.0363	0.2580	0.3487	
4	0.0653	0.3108	0.4785	
5	0.0083	0.3217	0.1593	

6	0.2173	0.3428	-1.0572
7	0.7068	0.7273	1.6985
8	38.8803	0.9976	-11.3456

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	0.9913	0.9418	1.0408	0.9162	1.0664
2	0.9450	0.9156	0.9744	0.8813	1.0086
3	0.9048	0.8761	0.9334	0.8415	0.9681
4	0.8699	0.8384	0.9013	0.8053	0.9345
5	0.8396	0.8076	0.8716	0.7747	0.9045
6	0.7707	0.7377	0.8037	0.7053	0.8361
7	0.7257	0.6775	0.7738	0.6515	0.7998
8	0.6613	0.6050	0.7177	0.5816	0.7411

2D Graph 21

LACTOSE ANHYDROUS (BiPhasic First-order reaction)

25°C / 60%RH

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.376037}}
b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.178844}}
c = yatxnear0(y,x)/2 "Auto {{previous: 0.615759}}
d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 0.000567885}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.98365048 Rsqr = 0.96756827 Adj Rsqr = 0.94324447

Standard Error of Estimate = 0.0311

	Coefficient		Std. Error	t	P
a	0.3760	0.2924	1.2861	0.2678	
b	0.1788	0.1569	1.1400	0.3179	
c	0.6158	0.3033	2.0301	0.1122	
d	0.0006	0.0222	0.0255	0.9809	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.1151	0.0384	39.7787	0.0020
Residual	4	0.0039	0.0010		
Total	7	0.1190	0.0170		

PRESS = 0.1794

Durbin-Watson Statistic = 3.4492

Normality Test: Passed (P = 0.7641)

Constant Variance Test: Passed (P = 0.4979)

Power of performed test with alpha = 0.0500: 0.9997

Regression Diagnostics:

Row	Predicted Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
-----	--------------------	-----------	------------	-----------------

1	0.9918	0.0082	0.2641	0.5747	0.5196
2	0.9299	-0.0340	-1.0947	-1.2839	-1.4501
3	0.8780	0.0356	1.1465	1.3412	1.5658
4	0.8346	-0.0095	-0.3066	-0.3700	-0.3260
5	0.7982	0.0129	0.4147	0.4999	0.4471
6	0.7208	-0.0287	-0.9229	-1.1457	-1.2106
7	0.6752	0.0169	0.5455	0.9895	0.9860
8	0.6193	-0.0014	-0.0467	-0.7843	-0.7384

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	0.3084	0.7888	1.0042
2	0.1548	0.2730	-0.8887
3	0.1657	0.2693	0.9506
4	0.0156	0.3134	-0.2202
5	0.0283	0.3119	0.3010
6	0.1776	0.3511	-0.8905
7	0.5606	0.6961	1.4923
8	43.3147	0.9965	-12.3919

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	0.9918	0.9152	1.0684	0.8764	1.1071
2	0.9299	0.8848	0.9749	0.8326	1.0272
3	0.8780	0.8333	0.9228	0.7809	0.9752
4	0.8346	0.7863	0.8829	0.7358	0.9334
5	0.7982	0.7501	0.8464	0.6995	0.8970
6	0.7208	0.6697	0.7719	0.6206	0.8211
7	0.6752	0.6032	0.7471	0.5628	0.7875
8	0.6193	0.5332	0.7054	0.4975	0.7412

2D Graph 22

LACTOSE ANHYDROUS (BiPhasic First-order reaction)

40°C / 75%RH

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.232507}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 1.80374}}

c = yatxnear0(y,x)/2 "Auto {{previous: 0.766668}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 0.0170271}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.98001052

Rsqr = 0.96042061

Adj Rsqr = 0.93073607

Standard Error of Estimate = 0.0351

	Coefficient		Std. Error	t	P
a	0.2325	0.0456	5.1038	0.0070	
b	1.8037	1.1450	1.5753	0.1903	
c	0.7667	0.0297	25.8543	<0.0001	
d	0.0170	0.0042	4.0412	0.0156	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.1194	0.0398	32.3542	0.0029
Residual	4	0.0049	0.0012		
Total	7	0.1244	0.0178		

PRESS = 3.2448

Durbin-Watson Statistic = 1.5293

Normality Test: Passed (P = 0.3720)

Constant Variance Test: Passed (P = 0.1597)

Power of performed test with alpha = 0.0500: 0.9993

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	0.9992	0.0008	0.0235	1.0960	1.1347
2	0.7920	-0.0090	-0.2575	-0.9725	-0.9639
3	0.7473	0.0183	0.5230	0.6052	0.5499
4	0.7295	0.0382	1.0899	1.3036	1.4887
5	0.7164	-0.0081	-0.2312	-0.2741	-0.2396
6	0.6805	-0.0491	-1.3992	-1.5632	-2.1704
7	0.6466	-0.0115	-0.3276	-0.3708	-0.3268
8	0.5454	0.0208	0.5943	1.3600	1.6063

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	651.5372	0.9995	52.8535
2	3.1371	0.9299	-3.5108
3	0.0311	0.2533	0.3203
4	0.1830	0.3011	0.9770
5	0.0076	0.2887	-0.1527
6	0.1516	0.1988	-1.0811
7	0.0097	0.2196	-0.1733
8	1.9589	0.8090	3.3063

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	0.9992	0.9018	1.0965	0.8615	1.1369
2	0.7920	0.6981	0.8859	0.6567	0.9273
3	0.7473	0.6983	0.7963	0.6383	0.8563
4	0.7295	0.6761	0.7830	0.6184	0.8406
5	0.7164	0.6640	0.7687	0.6058	0.8269
6	0.6805	0.6371	0.7240	0.5739	0.7872
7	0.6466	0.6010	0.6923	0.5391	0.7542
8	0.5454	0.4578	0.6330	0.4144	0.6764

2D Graph 23

LACTOSE ANHYDROUS (BiPhasic First-order reaction)

50°C / 20%moisture

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2. "Auto {{previous: 0.437135}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.915575}}

c = yatxnear0(y,x)/2 "Auto {{previous: 0.567457}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 0.00496292}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1
iterations=100

R = 0.99237002 Rsqr = 0.98479826 Adj Rsqr = 0.97339695

Standard Error of Estimate = 0.0267

	Coefficient		Std. Error	t	P
a	0.4371	0.0378	11.5746	0.0003	
b	0.9156	0.1858	4.9281	0.0079	
c	0.5675	0.0294	19.3051	<0.0001	
d	0.0050	0.0044	1.1327	0.3206	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.1852	0.0617	86.3759	0.0004
Residual	4	0.0029	0.0007		
Total	7	0.1880	0.0269		

PRESS = 0.0903

Durbin-Watson Statistic = 2.9064

Normality Test: Failed (P = 0.0080)

Constant Variance Test: Passed (P = 0.8849)

Power of performed test with alpha = 0.0500: 1.0000

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0046	-0.0046	-0.1718	-1.3469	-1.5779
2	0.7396	0.0202	0.7542	1.2487	1.3843
3	0.6319	-0.0164	-0.6129	-0.7524	-0.7033
4	0.5871	-0.0063	-0.2340	-0.2685	-0.2347
5	0.5675	-0.0202	-0.7543	-0.9040	-0.8776
6	0.5488	0.0403	1.5069	1.8406	4.0744
7	0.5400	-0.0086	-0.3227	-0.3760	-0.3315
8	0.5138	-0.0044	-0.1659	-0.5439	-0.4895

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	27.4338	0.9837	-12.2720	
2	0.6787	0.6352	1.8266	
3	0.0718	0.3365	-0.5008	
4	0.0057	0.2409	-0.1322	

5	0.0891	0.3037	-0.5796
6	0.4167	0.3297	2.8578
7	0.0126	0.2633	-0.1982
8	0.7211	0.9070	-1.5285

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0046	0.9310	1.0782	0.9001	1.1091
2	0.7396	0.6805	0.7988	0.6447	0.8345
3	0.6319	0.5888	0.6750	0.5461	0.7177
4	0.5871	0.5507	0.6235	0.5044	0.6698
5	0.5675	0.5266	0.6084	0.4828	0.6523
6	0.5488	0.5062	0.5914	0.4632	0.6344
7	0.5400	0.5019	0.5781	0.4566	0.6235
8	0.5138	0.4432	0.5845	0.4113	0.6163

2D Graph 24

LACTOSE ANHYDROUS (BiPhasic First-order reaction)

50°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatznear0(y,x)/2 "Auto {{previous: 0.574083}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.246131}}

c = yatznear0(y,x)/2 "Auto {{previous: 0.415514}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 1.41089e-010}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.91174814 Rsqr = 0.83128468 Adj Rsqr = 0.70474819

Standard Error of Estimate = 0.1190

	Coefficient		Std. Error	t	P
a	0.5741	0.5398	1.0635	0.3475	
b	0.2461	0.3295	0.7469	0.4966	
c	0.4155	0.5683	0.7312	0.5052	
d	0.0000	0.0714	0.0000	1.0000	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.2791	0.0930	6.5695	0.0503
Residual	4	0.0567	0.0142		
Total	7	0.3358	0.0480		

PRESS = 8.7426

Durbin-Watson Statistic = 2.7419

Normality Test: Passed (P = 0.0932)

Constant Variance Test: Passed (P = 0.7941)

Power of performed test with alpha = 0.0500: 0.9304

Regression Diagnostics:

Row	Predicted Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
-----	--------------------	-----------	------------	-----------------

1	0.9896	0.0104	0.0874	0.2068	0.1801
2	0.8643	0.0243	0.2045	0.2408	0.2101
3	0.7664	-0.1052	-0.8840	-1.0500	-1.0684
4	0.6899	-0.0401	-0.3373	-0.4072	-0.3602
5	0.6300	0.1879	1.5792	1.8776	4.7216
6	0.5180	-0.0866	-0.7277	-0.9174	-0.8942
7	0.4645	-0.0097	-0.0813	-0.1350	-0.1172
8	0.4197	0.0190	0.1593	1.9827	13.0744

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.0492	0.8214	0.3863	
2	0.0056	0.2792	0.1307	
3	0.1132	0.2911	-0.6847	
4	0.0190	0.3139	-0.2436	
5	0.3647	0.2927	3.0372	
6	0.1241	0.3709	-0.6865	
7	0.0080	0.6373	-0.1554	
8	151.2061	0.9935	162.1749	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	0.9896	0.6901	1.2891	0.5436	1.4355
2	0.8643	0.6898	1.0389	0.4906	1.2381
3	0.7664	0.5881	0.9447	0.3910	1.1419
4	0.6899	0.5047	0.8750	0.3111	1.0686
5	0.6300	0.4512	0.8088	0.2543	1.0057
6	0.5180	0.3168	0.7192	0.1311	0.9049
7	0.4645	0.2007	0.7283	0.0417	0.8873
8	0.4197	0.0903	0.7491	-0.0469	0.8862

2D Graph 25

.....
STARCH (First-order reaction)

25°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y = 1/abs(y)

reciprocal_ysquare = 1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q) = max(abs(q)) - abs(q)

yatxnear0(q,r) = xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 99.589}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.0057966}}

[Equation]

f = a * exp(-b * x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b > 0

[Options]

tolerance = 0.0001

stepsize = 100

iterations = 100

R = 0.88627805 Rsqr = 0.78548878 Adj Rsqr = 0.75484432

Standard Error of Estimate = 1.8803

	Coefficient		Std. Error	t	P
a	99.5890	0.9211	108.1172	<0.0001	
b	0.0058	0.0012	4.9607	0.0016	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	90.6282	90.6282	25.6323	0.0015
Residual	7	24.7499	3.5357		
Total	8	115.3781	14.4223		

PRESS = 35.3794

Durbin-Watson Statistic = 2.4030

Normality Test: Passed ($P = 0.5111$)

Constant Variance Test: Passed ($P = 0.8437$)

Power of performed test with $\alpha = 0.0500$: 0.9305

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	99.5890	0.4110	0.2186	0.2507	0.2332
2	99.0134	1.3790	0.7334	0.8191	0.7975
3	98.4411	1.3947	0.7417	0.8118	0.7897
4	97.8722	-2.2145	-1.1777	-1.2701	-1.3405
5	97.3065	-0.3822	-0.2032	-0.2170	-0.2016
6	96.1849	-0.4487	-0.2386	-0.2531	-0.2354
7	95.0762	-2.9799	-1.5848	-1.6971	-2.0481
8	93.9804	2.5533	1.3579	1.4916	1.6719
9	88.6876	0.2894	0.1539	0.2922	0.2721

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.0099	0.2400	0.1310	
2	0.0830	0.1984	0.3968	
3	0.0652	0.1653	0.3514	
4	0.1316	0.1403	-0.5414	
5	0.0033	0.1231	-0.0755	
6	0.0040	0.1114	-0.0833	
7	0.2115	0.1280	-0.7848	
8	0.2297	0.1712	0.7598	
9	0.1111	0.7224	0.4390	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	99.5890	97.4109	101.7671	94.6379	104.5402
2	99.0134	97.0329	100.9940	94.1459	103.8809
3	98.4411	96.6336	100.2487	93.6415	103.2408
4	97.8722	96.2070	99.5373	93.1243	102.6201
5	97.3065	95.7464	98.8665	92.5944	102.0185
6	96.1849	94.7011	97.6687	91.4975	100.8723
7	95.0762	93.4853	96.6672	90.3539	99.7986
8	93.9804	92.1408	95.8200	89.1685	98.7922
9	88.6876	84.9085	92.4667	82.8522	94.5229

2D Graph 27

STARCH (First-order reaction)

25°C / 60 %RH

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y = 1/abs(y)

reciprocal_ysquare = 1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q) = max(abs(q)) - abs(q)

yatxnear0(q,r) = xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 100.491}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.0037451}}

[Equation]

f = a * exp(-b * x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b > 0

[Options]

tolerance = 0.0001

stepsize = 100

iterations = 100

R = 0.72053762 Rsqr = 0.51917447 Adj Rsqr = 0.45048510

Standard Error of Estimate = 2.3001

	Coefficient		Std. Error	t	P
a	100.4908	1.1173	89.9384	<0.0001	
b	0.0037	0.0014	2.7187	0.0298	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	39.9875	39.9875	7.5583	0.0285
Residual	7	37.0338	5.2905		
Total	8	77.0213	9.6277		

PRESS = 57.1891

Durbin-Watson Statistic = 1.5556

Normality Test: Passed (P = 0.2828)

Constant Variance Test: Passed (P = 0.6116)

Power of performed test with alpha = 0.0500: 0.6049

The power of the performed test (0.6049) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	100.4908	-0.4908	-0.2134	-0.2441	-0.2270
2	100.1152	1.5850	0.6891	0.7687	0.7438
3	99.7410	4.1537	1.8059	1.9758	2.7505
4	99.3681	-1.7579	-0.7643	-0.8243	-0.8031
5	98.9967	-2.4960	-1.0852	-1.1591	-1.1937
6	98.2579	-1.5653	-0.6805	-0.7218	-0.6946
7	97.5247	-1.5963	-0.6940	-0.7426	-0.7163
8	96.7970	1.5069	0.6552	0.7184	0.6911
9	93.2389	0.6621	0.2878	0.5570	0.5275

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.0092	0.2360	-0.1262	
2	0.0722	0.1964	0.3678	
3	0.3847	0.1646	1.2210	
4	0.0555	0.1404	-0.3245	
5	0.0946	0.1235	-0.4481	

6	0.0326	0.1112	-0.2457
7	0.0400	0.1266	-0.2727
8	0.0522	0.1684	0.3109
9	0.4257	0.7329	0.8739

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	100.4908	97.8488	103.1329	94.4442	106.5375
2	100.1152	97.7045	102.5259	94.1660	106.0644
3	99.7410	97.5341	101.9478	93.8714	105.6105
4	99.3681	97.3304	101.4058	93.5600	105.1762
5	98.9967	97.0854	100.9079	93.2317	104.7616
6	98.2579	96.4441	100.0718	92.5245	103.9913
7	97.5247	95.5896	99.4598	91.7518	103.2976
8	96.7970	94.5653	99.0286	90.9180	102.6759
9	93.2389	88.5825	97.8952	86.0790	100.3987

2D Graph 28

STARCH (First-order reaction)

40°C / 75 %RH Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

```

a = yatknear0(y,x) "Auto {{previous: 101.175}}
b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.000653841}}
[Equation]
f=a*exp(-b*x)
fit f to y
"fit f to y with weight reciprocal_y
"fit f to y with weight reciprocal_ysquare
[Constraints]
b>0
[Options]
tolerance=0.0001
stepsize=100
iterations=100

```

R = 0.28113767 Rsqr = 0.07903839 Adj Rsqr = 0.00000000

Standard Error of Estimate = 1.4853

	Coefficient		Std. Error	t	P
a	101.1749	0.7123	142.0301	<0.0001	
b	0.0007	0.0008	0.7721	0.4653	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	1.3254	1.3254	0.6008	0.4637
Residual	7	15.4434	2.2062		
Total	8	16.7688	2.0961		

PRESS = 23.1707

Durbin-Watson Statistic = 2.9328

Normality Test: Passed (P = 0.7798)

Constant Variance Test: Passed (P = 0.4620)

Power of performed test with alpha = 0.0500: 0.1052

The power of the performed test (0.1052) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	101.1749	-1.1749	-0.7910	-0.9014	-0.8877
2	101.1088	-0.2626	-0.1768	-0.1968	-0.1827
3	101.0427	2.3228	1.5639	1.7100	2.0747

4	100.9766	-1.0661	-0.7178	-0.7742	-0.7496
5	100.9106	0.6437	0.4334	0.4631	0.4354
6	100.7788	-1.9748	-1.3295	-1.4102	-1.5430
7	100.6471	1.7310	1.1654	1.2455	1.3070
8	100.5155	0.1428	0.0961	0.1051	0.0974
9	99.8605	-0.3619	-0.2437	-0.4858	-0.4576

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.1214	0.2300	-0.4852	
2	0.0046	0.1934	-0.0895	
3	0.2860	0.1636	0.9177	
4	0.0490	0.1405	-0.3031	
5	0.0152	0.1241	0.1639	
6	0.1243	0.1111	-0.5455	
7	0.1104	0.1246	0.4931	
8	0.0011	0.1642	0.0432	
9	0.3511	0.7484	-0.7893	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	101.1749	99.4905	102.8593	97.2796	105.0702
2	101.1088	99.5640	102.6536	97.2718	104.9457
3	101.0427	99.6220	102.4634	97.2540	104.8314
4	100.9766	99.6601	102.2932	97.2258	104.7275
5	100.9106	99.6736	102.1477	97.1869	104.6344
6	100.7788	99.6080	101.9495	97.0765	104.4810
7	100.6471	99.4074	101.8867	96.9225	104.3717
8	100.5155	99.0921	101.9389	96.7258	104.3053
9	99.8605	96.8220	102.8990	95.2163	104.5047

2D Graph 29

.....
STARCH (First-order reaction)

50°C / 20 %moisture

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 91.2028}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.0200502}}

[Equation]

f=a*exp(-b*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

[Options]

tolerance=0.0001

stepsize=100

iterations=100

R = 0.68798381 Rsqr = 0.47332173 Adj Rsqr = 0.39808197

Standard Error of Estimate = 9.8936

	Coefficient		Std. Error	t	P
a	91.2028	5.1320	17.7715	<0.0001	
b	0.0201	0.0081	2.4673	0.0430	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	615.7738	615.7738	6.2908	0.0405
Residual	7	685.1887	97.8841		
Total	8	1300.9624	162.6203		

PRESS = 2008.9507

Durbin-Watson Statistic = 0.7846

Normality Test: Passed ($P = 0.7679$)

Constant Variance Test: Passed ($P = 0.4905$)

Power of performed test with $\alpha = 0.0500$: 0.5429

The power of the performed test (0.5429) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	91.2028	8.7972	0.8892	1.0400	1.0472
2	89.3924	7.8634	0.7948	0.8952	0.8808
3	87.6179	2.8554	0.2886	0.3166	0.2952
4	85.8786	0.1941	0.0196	0.0211	0.0196
5	84.1739	-3.8098	-0.3851	-0.4107	-0.3849
6	80.8653	-16.4729	-1.6650	-1.7692	-2.2030
7	77.6867	-9.1857	-0.9284	-1.0016	-1.0019
8	74.6331	-2.4393	-0.2466	-0.2744	-0.2554
9	61.0737	12.7112	1.2848	2.1489	3.4105

Influence Diagnostics:

Row	Cook's	Dist	Leverage	DFFITS
1	0.1991	0.2691	0.6354	
2	0.1077	0.2118	0.4566	
3	0.0102	0.1689	0.1331	
4	0.0000	0.1391	0.0079	
5	0.0116	0.1208	-0.1427	
6	0.2021	0.1144	-0.7917	
7	0.0822	0.1408	-0.4056	
8	0.0090	0.1926	-0.1248	
9	4.1505	0.6425	4.5725	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	91.2028	79.0676	103.3379	64.8480	117.5576
2	89.3924	78.6258	100.1589	63.6390	115.1457
3	87.6179	78.0020	97.2337	62.3240	112.9117
4	85.8786	77.1545	94.6027	60.9102	110.8471
5	84.1739	76.0423	92.3055	59.4062	108.9415
6	80.8653	72.9535	88.7770	56.1689	105.5616
7	77.6867	68.9082	86.4651	52.6992	102.6742
8	74.6331	64.3657	84.9004	49.0844	100.1817
9	61.0737	42.3208	79.8267	31.0906	91.0569

2D Graph 30

STARCH (First-order reaction)

50°C

Nonlinear Regression

[Variables]

$x = \text{col}(1)$

$y = \text{col}(2)$

$\text{reciprocal_y} = 1/\text{abs}(y)$

$\text{reciprocal_ysquare} = 1/y^2$

'Automatic Initial Parameter Estimate Functions

$\text{xnear0}(q) = \max(\text{abs}(q)) - \text{abs}(q)$

$\text{yatznear0}(q,r) = \text{xatymax}(q, \text{xnear0}(r))$

[Parameters]

$a = \text{yatznear0}(y,x)$ "Auto {{previous: 98.2956}}

$b = -\ln(.5)/(\text{x50}(x,y) - \min(x))$ "Auto {{previous: 0.0556155}}

[Equation]

$f = a * \exp(-b * x)$

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

$b > 0$

[Options]

tolerance=0.0001

stepsize=100

iterations=100

$R = 0.97292605$

$\text{Rsqr} = 0.94658509$

$\text{Adj Rsqr} = 0.93895439$

Standard Error of Estimate = 5.0520

	Coefficient		Std. Error	t	P
a	98.2956	2.9874	32.9031	<0.0001	
b	0.0556	0.0059	9.3630	<0.0001	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	3166.0193	3166.0193	124.0496	<0.0001
Residual	7	178.6555	25.5222		
Total	8	3344.6748	418.0844		

PRESS = 366.1160

Durbin-Watson Statistic = 1.1962

Normality Test: Passed (P = 0.5334)

Constant Variance Test: Failed (P = 0.0361)

Power of performed test with alpha = 0.0500: 0.9995

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	98.2956	1.7044	0.3374	0.4184	0.3923
2	92.9781	1.6679	0.3302	0.3789	0.3545
3	87.9482	1.6226	0.3212	0.3531	0.3298
4	83.1905	4.0165	0.7950	0.8548	0.8362
5	78.6901	-2.6386	-0.5223	-0.5569	-0.5274
6	70.4066	-7.3276	-1.4504	-1.5614	-1.7907
7	62.9950	-3.4158	-0.6761	-0.7495	-0.7236
8	56.3637	-2.5136	-0.4975	-0.5728	-0.5432
9	32.3195	8.6927	1.7207	2.2455	3.9314

Influence Diagnostics:

Row	Cook's Dist		Leverage	DFITS
1	0.0471	0.3497	0.2876	
2	0.0228	0.2408	0.1997	
3	0.0130	0.1724	0.1505	
4	0.0569	0.1349	0.3301	
5	0.0212	0.1204	-0.1951	
6	0.1937	0.1371	-0.7138	
7	0.0643	0.1863	-0.3462	
8	0.0534	0.2455	-0.3099	
9	1.7729	0.4129	3.2967	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	98.2956	91.2315	105.3598	84.4173	112.1740
2	92.9781	87.1155	98.8407	79.6711	106.2851
3	87.9482	82.9879	92.9086	75.0134	100.8831
4	83.1905	78.8036	87.5774	70.4645	95.9165
5	78.6901	74.5448	82.8354	66.0454	91.3348
6	70.4066	65.9831	74.8300	57.6679	83.1452
7	62.9950	57.8392	68.1509	49.9839	76.0061
8	56.3637	50.4442	62.2831	43.0315	69.6958
9	32.3195	24.6437	39.9952	18.1200	46.5189

2D Graph 31

STARCH (BiPhasic First-order reaction)

25°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y = 1/abs(y)

reciprocal_ysquare = 1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q) = max(abs(q)) - abs(q)

yatxnear0(q,r) = xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.0275784}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.361565}}

c = yatznear0(y,x)/2 "Auto {{previous: 0.979507}}
d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 0.00457035}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.90054905 Rsqr = 0.81098859 Adj Rsqr = 0.69758174

Standard Error of Estimate = 0.0209

	Coefficient		Std. Error	t	P
a	0.0276	0.0485	0.5682	0.5945	
b	0.3616	1.1458	0.3156	0.7651	
c	0.9795	0.0498	19.6638	<0.0001	
d	0.0046	0.0033	1.4037	0.2194	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.0094	0.0031	7.1511	0.0294
Residual	5	0.0022	0.0004		
Total	8	0.0115	0.0014		

PRESS = 0.0580

Durbin-Watson Statistic = 2.9669

Normality Test: Passed (P = 0.7524)

Constant Variance Test: Passed (P = 0.7418)

Power of performed test with alpha = 0.0500: 0.9509

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0071	-0.0071	-0.3393	-0.9380	-0.9243
2	0.9943	0.0097	0.4631	0.5547	0.5121
3	0.9840	0.0144	0.6886	0.8374	0.8078

4	0.9755	-0.0189	-0.9056	-1.0750	-1.0965
5	0.9683	0.0010	0.0472	0.0540	0.0483
6	0.9562	0.0012	0.0574	0.0662	0.0592
7	0.9459	-0.0249	-1.1926	-1.4792	-1.7641
8	0.9365	0.0288	1.3813	1.7851	2.6514
9	0.8940	-0.0042	-0.2008	-1.4659	-1.7363

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	1.4614	0.8692	-2.3823	
2	0.0334	0.3028	0.3375	
3	0.0839	0.3237	0.5589	
4	0.1181	0.2902	-0.7012	
5	0.0002	0.2344	0.0267	
6	0.0004	0.2471	0.0339	
7	0.2945	0.3500	-1.2945	
8	0.5340	0.4013	2.1707	
9	28.0975	0.9812	-12.5570	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0071	0.9570	1.0571	0.9337	1.0805
2	0.9943	0.9647	1.0238	0.9330	1.0555
3	0.9840	0.9534	1.0145	0.9222	1.0457
4	0.9755	0.9466	1.0044	0.9145	1.0365
5	0.9683	0.9423	0.9942	0.9086	1.0279
6	0.9562	0.9295	0.9829	0.8962	1.0161
7	0.9459	0.9141	0.9776	0.8835	1.0082
8	0.9365	0.9025	0.9705	0.8729	1.0000
9	0.8940	0.8408	0.9471	0.8184	0.9695

2D Graph 31

.....
STARCH (BiPhasic First-order reaction)

25°C / 60%RH

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.0463895}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.200272}}

c = yatxnear0(y,x)/2 "Auto {{previous: 0.970291}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 0.00152882}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.75880383

Rsqr = 0.57578325

Adj Rsqr = 0.32125321

Standard Error of Estimate = 0.0256

	Coefficient		Std. Error	t	P
a	0.0464	0.1746	0.2657	0.8011	
b	0.2003	0.9406	0.2129	0.8398	
c	0.9703	0.1830	5.3014	0.0032	
d	0.0015	0.0090	0.1694	0.8722	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.0044	0.0015	2.2621	0.1989
Residual	5	0.0033	0.0007		
Total	8	0.0077	0.0010		

PRESS = 20.5658

Durbin-Watson Statistic = 1.9522

Normality Test: Passed (P = 0.2124)

Constant Variance Test: Passed (P = 0.2852)

Power of performed test with alpha = 0.0500: 0.6820

The power of the performed test (0.6820) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0167	-0.0167	-0.6525	-1.4311	-1.6659
2	1.0068	0.0102	0.3999	0.4694	0.4294
3	0.9984	0.0405	1.5858	1.8609	3.0017
4	0.9913	-0.0152	-0.5941	-0.7130	-0.6728
5	0.9852	-0.0202	-0.7898	-0.9341	-0.9196
6	0.9754	-0.0085	-0.3307	-0.3770	-0.3421
7	0.9678	-0.0086	-0.3348	-0.4081	-0.3712
8	0.9618	0.0212	0.8296	1.1842	1.2486
9	0.9419	-0.0029	-0.1137	-4.4912	(+inf)

Influence Diagnostics:

Row	Cook's Dist		Leverage	DFFITS
1	1.9509	0.7921	-3.2518	
2	0.0208	0.2742	0.2639	
3	0.3263	0.2737	1.8429	
4	0.0560	0.3057	-0.4464	
5	0.0870	0.2851	-0.5807	
6	0.0107	0.2306	-0.1873	
7	0.0202	0.3270	-0.2588	
8	0.3636	0.5092	1.2717	
9	7857.7743	0.9994	(+inf)	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0167	0.9582	1.0752	0.9287	1.1046
2	1.0068	0.9724	1.0412	0.9326	1.0810
3	0.9984	0.9640	1.0328	0.9242	1.0726
4	0.9913	0.9550	1.0276	0.9162	1.0664
5	0.9852	0.9501	1.0203	0.9107	1.0597
6	0.9754	0.9438	1.0069	0.9025	1.0483

7	0.9678	0.9303	1.0054	0.8921	1.0435
8	0.9618	0.9149	1.0087	0.8811	1.0426
9	0.9419	0.8762	1.0076	0.8490	1.0348

2D Graph 32

STARCH (BiPhasic First-order reaction)

40°C / 75%RH

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.284321}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.000676696}}

c = yatxnear0(y,x)/2 "Auto {{previous: 0.727428}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 0.000644927}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0
 [Options]
 tolerance=1e-6
 stepsize=0.1
 iterations=100

R = 0.28113749 Rsqr = 0.07903829 Adj Rsqr = 0.00000000

Standard Error of Estimate = 0.0176

	Coefficient		Std. Error	t	P
a	0.2843	285635.0184	0.0000	1.0000	
b	0.0007	23.9808	0.0000	1.0000	
c	0.7274	285635.0184	0.0000	1.0000	
d	0.0006	9.3552	0.0001	0.9999	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.0001	0.0000	0.1430	0.9298
Residual	5	0.0015	0.0003		
Total	8	0.0017	0.0002		

PRESS = 0.0057

Durbin-Watson Statistic = 2.9328

Normality Test: Passed (P = 0.7798)

Constant Variance Test: Passed (P = 0.4620)

Power of performed test with alpha = 0.0500: 0.1052

The power of the performed test (0.1052) is below the desired power of 0.8000.
 You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0117	-0.0117	-0.6685	-0.8456	-0.8170
2	1.0111	-0.0026	-0.1494	-0.1742	-0.1563
3	1.0104	0.0232	1.3217	1.3850	1.5779
4	1.0098	-0.0107	-0.6066	-0.6344	-0.5918
5	1.0091	0.0064	0.3663	0.3854	0.3499
6	1.0078	-0.0197	-1.1236	-1.2081	-1.2841
7	1.0065	0.0173	0.9849	1.0946	1.1228
8	1.0052	0.0014	0.0812	0.1021	0.0914
9	0.9986	-0.0036	-0.2059	-0.8329	-0.8027

Influence Diagnostics:

Row	Cook's Dist		Leverage	DFFITS
1	0.1073	0.3750	-0.6329	
2	0.0027	0.2647	-0.0938	
3	0.0470	0.0893	0.4940	
4	0.0094	0.0857	-0.1812	
5	0.0040	0.0967	0.1145	
6	0.0569	0.1349	-0.5070	
7	0.0704	0.1904	0.5445	
8	0.0015	0.3667	0.0696	
9	2.6632	0.9389	-3.1456	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0117	0.9841	1.0394	0.9588	1.0647
2	1.0111	0.9878	1.0343	0.9603	1.0619
3	1.0104	0.9969	1.0239	0.9633	1.0576
4	1.0098	0.9965	1.0230	0.9627	1.0568
5	1.0091	0.9951	1.0232	0.9618	1.0564
6	1.0078	0.9912	1.0244	0.9597	1.0559
7	1.0065	0.9868	1.0262	0.9572	1.0558
8	1.0052	0.9778	1.0325	0.9523	1.0580
9	0.9986	0.9548	1.0424	0.9357	1.0615

2D Graph 33

STARCH (BiPhasic First-order reaction)
50°C / 20 %moisture
Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.346976}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.307337}}

c = yatxnear0(y,x)/2 "Auto {{previous: 0.68886}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 2.12148e-010}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.92855729 Rsqr = 0.86221864 Adj Rsqr = 0.77954983

Standard Error of Estimate = 0.0599

	Coefficient		Std. Error	t	P
a	0.3470	0.1666	2.0828	0.0917	
b	0.3073	0.2517	1.2208	0.2766	
c	0.6889	0.1751	3.9336	0.0110	
d	0.0000	0.0149	0.0000	1.0000	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.1122	0.0374	10.4298	0.0136
Residual	5	0.0179	0.0036		
Total	8	0.1301	0.0163		

PRESS = 16.6152

Durbin-Watson Statistic = 1.4125

Normality Test: Failed (P = 0.0446)

Constant Variance Test: Passed (P = 0.8094)

Power of performed test with alpha = 0.0500: 0.9811

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0358	-0.0358	-0.5985	-1.5159	-1.8443
2	0.9440	0.0285	0.4765	0.5644	0.5217
3	0.8765	0.0282	0.4714	0.5648	0.5221
4	0.8269	0.0339	0.5657	0.6739	0.6322
5	0.7903	0.0133	0.2221	0.2560	0.2305
6	0.7437	-0.0998	-1.6672	-1.9075	-3.2699
7	0.7185	-0.0335	-0.5600	-0.6951	-0.6542
8	0.7049	0.0170	0.2843	0.3823	0.3470
9	0.6896	0.0482	0.8058	7.3977	(+inf)

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	3.1104	0.8441	-4.2915	
2	0.0321	0.2872	0.3311	
3	0.0348	0.3036	0.3447	
4	0.0476	0.2955	0.4094	
5	0.0054	0.2477	0.1323	
6	0.2812	0.2361	-1.8181	
7	0.0653	0.3509	-0.4810	
8	0.0295	0.4468	0.3119	
9	1139.4517	0.9881	(+inf)	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0358	0.8944	1.1772	0.8268	1.2448
2	0.9440	0.8615	1.0265	0.7694	1.1186
3	0.8765	0.7917	0.9613	0.7008	1.0522
4	0.8269	0.7432	0.9105	0.6517	1.0020
5	0.7903	0.7137	0.8669	0.6184	0.9623
6	0.7437	0.6690	0.8185	0.5726	0.9149
7	0.7185	0.6274	0.8097	0.5397	0.8974
8	0.7049	0.6020	0.8078	0.5198	0.8900
9	0.6896	0.5366	0.8426	0.4726	0.9066

2D Graph 34

STARCH (BiPhasic First-order reaction)

50°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y = 1/abs(y)

reciprocal_ysquare = 1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q) = max(abs(q)) - abs(q)

yatxnear0(q,r) = xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.687449}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.12064}}

c = yatxnear0(y,x)/2 "Auto {{previous: 0.338624}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 6.66952e-010}}

[Equation]

f = a*exp(-b*x) + c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b > 0

d > 0

[Options]

tolerance = 1e-6

stepsize = 0.1

iterations=100

R = 0.99125473 Rsqr = 0.98258593 Adj Rsqr = 0.97213749

Standard Error of Estimate = 0.0341

	Coefficient		Std. Error	t	P
a	0.6874	0.8355	0.8228	0.4481	
b	0.1206	0.1314	0.9178	0.4008	
c	0.3386	0.8503	0.3982	0.7069	
d	0.0000	0.0922	0.0000	1.0000	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.3286	0.1095	94.0414	<0.0001
Residual	5	0.0058	0.0012		
Total	8	0.3345	0.0418		

PRESS = 19.8656

Durbin-Watson Statistic = 1.4971

Normality Test: Passed (P = 0.4066)

Constant Variance Test: Passed (P = 0.7755)

Power of performed test with alpha = 0.0500: 1.0000

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0261	-0.0261	-0.7639	-1.5218	-1.8577
2	0.9479	-0.0015	-0.0435	-0.0509	-0.0456
3	0.8787	0.0170	0.4984	0.5719	0.5291
4	0.8173	0.0548	1.6042	1.8955	3.1957
5	0.7629	-0.0024	-0.0704	-0.0835	-0.0747
6	0.6720	-0.0412	-1.2062	-1.3829	-1.5741
7	0.6005	-0.0047	-0.1379	-0.1682	-0.1509
8	0.5444	-0.0059	-0.1716	-0.2721	-0.2452
9	0.4002	0.0099	0.2909	6.1616	(+inf)

Influence Diagnostics:

Row	Cook's Dist		Leverage	DFFITS
1	1.7186	0.7480	-3.2006	
2	0.0002	0.2706	-0.0278	
3	0.0259	0.2405	0.2977	
4	0.3559	0.2838	2.0115	

5	0.0007	0.2895	-0.0477
6	0.1504	0.2393	-0.8829
7	0.0035	0.3281	-0.1054
8	0.0281	0.6024	-0.3019
9	4249.5856	0.9978	(+inf)

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0261	0.9502	1.1020	0.9101	1.1421
2	0.9479	0.9023	0.9936	0.8491	1.0468
3	0.8787	0.8357	0.9217	0.7810	0.9764
4	0.8173	0.7706	0.8641	0.7179	0.9167
5	0.7629	0.7157	0.8101	0.6633	0.8625
6	0.6720	0.6290	0.7149	0.5743	0.7696
7	0.6005	0.5502	0.6508	0.4994	0.7016
8	0.5444	0.4763	0.6125	0.4333	0.6554
9	0.4002	0.3126	0.4878	0.2762	0.5242

2D Graph 35

.....
TALC (First-order reaction)

25°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

Automatic Initial Parameter Estimate Functions

$x_{near0}(q) = \max(\text{abs}(q)) - \text{abs}(q)$

$y_{atxnear0}(q,r) = x_{atymax}(q, x_{near0}(r))$

[Parameters]

$a = y_{atxnear0}(y,x)$ "Auto {{previous: 102.241}}

$b = -\ln(.5)/(x_{50}(x,y) - \min(x))$ "Auto {{previous: 0.0129317}}

[Equation]

$f = a * \exp(-b * x)$

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

$b > 0$

[Options]

tolerance=0.0001

stepsize=100

iterations=100

$R = 0.94669224$ $R_{sqr} = 0.89622621$ $Adj\ R_{sqr} = 0.88140138$

Standard Error of Estimate = 2.6157

	Coefficient		Std. Error	t	P
a	102.2411	1.3191	77.5072	<0.0001	
b	0.0129	0.0017	7.4144	0.0001	

Analysis of Variance:

	DF	SS	MS	F	P
Regression 1		413.6188	413.6188	60.4544	0.0001
Residual 7		47.8928	6.8418		
Total 8		461.5116	57.6890		

PRESS = 81.3825

Durbin-Watson Statistic = 2.2603

Normality Test: Passed (P = 0.7689)

Constant Variance Test: Passed (P = 0.2230)

Power of performed test with alpha = 0.0500: 0.9928

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	102.2411	-2.2411	-0.8568	-0.9922	-0.9909
2	100.9275	2.5643	0.9804	1.0997	1.1194

3	99.6308	3.4156	1.3058	1.4310	1.5751
4	98.3507	-0.9936	-0.3798	-0.4095	-0.3838
5	97.0870	-2.0161	-0.7708	-0.8225	-0.8012
6	94.6082	1.9769	0.7558	0.8022	0.7794
7	92.1927	-3.7668	-1.4401	-1.5474	-1.7662
8	89.8389	-0.1307	-0.0500	-0.0552	-0.0511
9	78.9410	1.2093	0.4623	0.8219	0.8006

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.1679	0.2543	-0.5787	
2	0.1561	0.2052	0.5688	
3	0.2057	0.1673	0.7060	
4	0.0136	0.1397	-0.1547	
5	0.0469	0.1219	-0.2985	
6	0.0408	0.1124	0.2774	
7	0.1851	0.1339	-0.6944	
8	0.0003	0.1816	-0.0241	
9	0.7298	0.6836	1.1767	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	102.2411	99.1219	105.3604	95.3140	109.1683
2	100.9275	98.1255	103.7295	94.1373	107.7177
3	99.6308	97.1010	102.1605	92.9483	106.3132
4	98.3507	96.0386	100.6628	91.7475	104.9538
5	97.0870	94.9278	99.2462	90.5358	103.6382
6	94.6082	92.5342	96.6822	88.0846	101.1318
7	92.1927	89.9295	94.4559	85.6065	98.7789
8	89.8389	87.2029	92.4748	83.1155	96.5622
9	78.9410	73.8272	84.0549	70.9156	86.9664

2D Graph 32

TALC (First-order reaction)

25°C / 60%RH

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 101.384}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.00392561}}

[Equation]

f=a*exp(-b*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

[Options]

tolerance=0.0001

stepsize=100

iterations=100

R = 0.78307565 Rsqr = 0.61320747 Adj Rsqr = 0.55795139

Standard Error of Estimate = 2.0110

	Coefficient		Std. Error	t	P
a	101.3838	0.9776	103.7032	<0.0001	
b	0.0039	0.0012	3.2802	0.0135	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	44.8811	44.8811	11.0976	0.0126
Residual	7	28.3096	4.0442		
Total	8	73.1907	9.1488		

PRESS = 41.1859

Durbin-Watson Statistic = 2.0292

Normality Test: Passed (P = 0.7028)

Constant Variance Test: Passed (P = 0.9477)

Power of performed test with alpha = 0.0500: 0.7324

The power of the performed test (0.7324) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	101.3838	-1.3838	-0.6881	-0.7874	-0.7636
2	100.9866	1.6822	0.8365	0.9332	0.9234
3	100.5909	-0.7303	-0.3631	-0.3973	-0.3721
4	100.1968	-1.9726	-0.9809	-1.0579	-1.0686
5	99.8042	1.6896	0.8401	0.8974	0.8831
6	99.0237	2.9685	1.4761	1.5657	1.7983
7	98.2493	0.0724	0.0360	0.0385	0.0357
8	97.4810	-2.7063	-1.3457	-1.4759	-1.6464
9	93.7284	0.3804	0.1891	0.3654	0.3415

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.0959	0.2363	-0.4248	
2	0.1066	0.1966	0.4568	
3	0.0156	0.1647	-0.1652	
4	0.0914	0.1404	-0.4318	
5	0.0567	0.1234	0.3314	
6	0.1534	0.1112	0.6362	
7	0.0001	0.1267	0.0136	
8	0.2209	0.1686	-0.7414	
9	0.1823	0.7320	0.5645	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	101.3838	99.0720	103.6955	96.0963	106.6712
2	100.9866	98.8779	103.0952	95.7847	106.1884
3	100.5909	98.6611	102.5207	95.4589	105.7229
4	100.1968	98.4153	101.9783	95.1187	105.2749
5	99.8042	98.1334	101.4750	94.7639	104.8445
6	99.0237	97.4378	100.6097	94.0109	104.0365
7	98.2493	96.5566	99.9420	93.2017	103.2969
8	97.4810	95.5284	99.4336	92.3404	102.6216
9	93.7284	89.6598	97.7969	87.4701	99.9867

2D Graph 33

.....

TALC (First-order reaction)

40°C / 75%RH

NO OUTPUT

.....

TALC (First-order reaction)

50°C / 20%moisture

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 84.9833}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.0272392}}

[Equation]

f=a*exp(-b*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

[Options]

tolerance=0.0001
 stepsize=100
 iterations=100

R = 0.7685 Rsqr = 0.5906 Adj Rsqr = 0.5321

Standard Error of Estimate = 9.2954

	Coefficient		Std. Error	t	P
a	84.9833	4.9568	17.1447	<0.0001	
b	0.0272	0.0090	3.0307	0.0191	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	872.5961	872.5961	10.0989	0.0155
Residual	7	604.8367	86.4052		
Total	8	1477.4328	184.6791		

PRESS = 1511.2094

Durbin-Watson Statistic = 1.2288

Normality Test: Passed (P = 0.6711)

Constant Variance Test: Passed (P = 0.6758)

Power of performed test with alpha = 0.0500: 0.7021

The power of the performed test (0.7021) is below the desired power of 0.8000.
 You should interpret the negative findings cautiously..

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	84.9833	15.0167	1.6155	1.9097	2.5545
2	82.6997	4.4898	0.4830	0.5463	0.5169
3	80.4774	-1.3672	-0.1471	-0.1615	-0.1498
4	78.3149	-11.6707	-1.2555	-1.3525	-1.4569
5	76.2105	-2.7199	-0.2926	-0.3119	-0.2908
6	72.1697	-8.1631	-0.8782	-0.9347	-0.9250
7	68.3432	0.3788	0.0408	0.0442	0.0409
8	64.7196	-5.8232	-0.6265	-0.7021	-0.6742
9	49.2876	10.6308	1.1437	1.8062	2.2884

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.7245	0.2844	1.6102	

2	0.0416	0.2182	0.2731
3	0.0027	0.1703	-0.0679
4	0.1467	0.1383	-0.5836
5	0.0066	0.1200	-0.1074
6	0.0580	0.1172	-0.3370
7	0.0002	0.1487	0.0171
8	0.0632	0.2040	-0.3413
9	2.4371	0.5991	2.7971

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	84.9833	73.2623	96.7044	60.0733	109.8934
2	82.6997	72.4326	92.9668	58.4398	106.9596
3	80.4774	71.4074	89.5475	56.6994	104.2555
4	78.3149	70.1420	86.4878	54.8644	101.7654
5	76.2105	68.5966	83.8243	52.9489	99.4720
6	72.1697	64.6451	79.6943	48.9372	95.4022
7	68.3432	59.8670	76.8194	44.7853	91.9011
8	64.7196	54.7927	74.6465	40.6017	88.8375
9	49.2876	32.2752	66.3000	21.4927	77.0824

2D Graph 35

.....
TALC (First-order reaction)

50°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

```

reciprocal_y=1/abs(y)
reciprocal_ysquare=1/y^2
'Automatic Initial Parameter Estimate Functions
xnear0(q)=max(abs(q))-abs(q)
yatxnear0(q,r)=xatymax(q,xnear0(r))
[Parameters]
a = yatxnear0(y,x) "Auto {{previous: 98.224}}
b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.0510482}}
[Equation]
f=a*exp(-b*x)
fit f to y
"fit f to y with weight reciprocal_y
"fit f to y with weight reciprocal_ysquare
[Constraints]
b>0
[Options]
tolerance=0.0001
stepsize=100
iterations=100

```

R = 0.98210601 Rsqr = 0.96453222 Adj Rsqr = 0.95946539

Standard Error of Estimate = 3.9457

	Coefficient		Std. Error	t	P
a	98.2240	2.2977	42.7480	<0.0001	
b	0.0510	0.0044	11.5676	<0.0001	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	2963.6243	2963.6243	190.3622	<0.0001
Residual	7	108.9784	15.5683		
Total	8	3072.6027	384.0753		

PRESS = 198.1004

Durbin-Watson Statistic = 1.1975

Normality Test: Passed (P = 0.6137)

Constant Variance Test: Passed (P = 0.4905)

Power of performed test with alpha = 0.0500: 0.9999

Regression Diagnostics:

Row	Predicted Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
-----	--------------------	-----------	------------	-----------------

1	98.2240	1.7760	0.4501	0.5537	0.5242
2	93.3357	6.8087	1.7256	1.9764	2.7523
3	88.6906	-1.4945	-0.3788	-0.4163	-0.3903
4	84.2768	-4.6651	-1.1823	-1.2715	-1.3423
5	80.0826	-2.2969	-0.5821	-0.6205	-0.5909
6	72.3099	-3.4261	-0.8683	-0.9326	-0.9227
7	65.2917	-0.8959	-0.2271	-0.2508	-0.2332
8	58.9547	1.0517	0.2666	0.3058	0.2850
9	35.3850	4.0678	1.0310	1.3805	1.4982

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS	
1	0.0787	0.3391	0.3755		
2	0.6089	0.2377	1.5368		
3	0.0181	0.1724	-0.1781		
4	0.1265	0.1353	-0.5310		
5	0.0262	0.1199	-0.2181		
6	0.0668	0.1332	-0.3617		
7	0.0069	0.1801	-0.1093		
8	0.0148	0.2400	0.1602		
9	0.7556	0.4423	1.3342		

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	98.2240	92.7907	103.6573	87.4272	109.0208
2	93.3357	88.7871	97.8843	82.9559	103.7154
3	88.6906	84.8167	92.5646	78.5883	98.7930
4	84.2768	80.8446	87.7089	74.3355	94.2181
5	80.0826	76.8524	83.3127	70.2092	89.9559
6	72.3099	68.9051	75.7148	62.3781	82.2418
7	65.2917	61.3317	69.2518	55.1561	75.4274
8	58.9547	54.3837	63.5256	48.5651	69.3442
9	35.3850	29.1801	41.5898	24.1801	46.5899

2D Graph 36

TALC (BiPhasic First-order reaction)

25°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.411743}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.0412329}}

c = yatxnear0(y,x)/2 "Auto {{previous: 0.618745}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 2.52176e-011}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1
iterations=100

R = 0.95072798 Rsqr = 0.90388369 Adj Rsqr = 0.84621390

Standard Error of Estimate = 0.0298

	Coefficient		Std. Error	t	P
a	0.4117	15.0848	0.0273	0.9793	
b	0.0412	0.9213	0.0448	0.9660	
c	0.6187	15.0995	0.0410	0.9689	
d	0.0000	0.4174	0.0000	1.0000	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.0417	0.0139	15.6734	0.0056
Residual	5	0.0044	0.0009		
Total	8	0.0462	0.0058		

PRESS = 6.9041

Durbin-Watson Statistic = 2.4558

Normality Test: Passed (P = 0.7024)

Constant Variance Test: Passed (P = 0.1243)

Power of performed test with alpha = 0.0500: 0.9945

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
2	1.0305	-0.0305	-1.0236	-1.8784	-3.0970
3	1.0139	0.0211	0.7071	0.8292	0.7986
4	0.9979	0.0326	1.0934	1.2382	1.3300
5	0.9826	-0.0090	-0.3025	-0.3535	-0.3202
6	0.9679	-0.0172	-0.5766	-0.6879	-0.6466
7	0.9402	0.0256	0.8596	0.9980	0.9975
8	0.9148	-0.0305	-1.0253	-1.2382	-1.3301
9	0.8914	0.0057	0.1920	0.3328	0.3010
10	0.7992	0.0023	0.0758	2.5836	(+inf)

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
2	2.0886	0.7031	-4.7655	
3	0.0645	0.2728	0.4892	
4	0.1083	0.2202	0.7069	

5	0.0114	0.2679	-0.1937
6	0.0501	0.2974	-0.4207
7	0.0866	0.2581	0.5884
8	0.1758	0.3144	-0.9007
9	0.0555	0.6670	0.4260
10	1938.9740	0.9991	(+inf)

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
2	1.0305	0.9663	1.0947	0.9306	1.1304
3	1.0139	0.9739	1.0538	0.9275	1.1002
4	0.9979	0.9620	1.0338	0.9133	1.0825
5	0.9826	0.9430	1.0222	0.8964	1.0688
6	0.9679	0.9261	1.0096	0.8807	1.0551
7	0.9402	0.9013	0.9791	0.8544	1.0261
8	0.9148	0.8719	0.9577	0.8270	1.0026
9	0.8914	0.8288	0.9539	0.7925	0.9902
10	0.7992	0.7227	0.8758	0.6910	0.9075

2D Graph 36

TALC (BiPhasic First-order reaction)

25°C / 60%RH

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

$xnear0(q)=\max(\text{abs}(q))-\text{abs}(q)$

$yatxnear0(q,r)=xatymax(q,xnear0(r))$

[Parameters]

$a = yatxnear0(y,x)/2$ "Auto {{previous: 0.45219}}

$b = -\ln(.5)/(0.5*(x50(x,y)-\min(x)))$ "Auto {{previous: 0.00453869}}

$c = yatxnear0(y,x)/2$ "Auto {{previous: 0.561641}}

$d = -\ln(.5)/(1.5*(x50(x,y)-\min(x)))$ "Auto {{previous: 0.00342691}}

[Equation]

$f=a*\exp(-b*x)+c*\exp(-d*x)$

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

$b>0$

$d>0$

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

$R = 0.78307374$ $Rsqr = 0.61320448$ $Adj Rsqr = 0.38112716$

Standard Error of Estimate = 0.0238

	Coefficient		Std. Error	t	P
a	0.4522	736579.2042	0.0000	1.0000	
b	0.0045	358.6938	0.0000	1.0000	
c	0.5616	736579.2032	0.0000	1.0000	
d	0.0034	289.4781	0.0000	1.0000	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.0045	0.0015	2.6422	0.1610
Residual	5	0.0028	0.0006		
Total	8	0.0073	0.0009		

PRESS = 0.0134

Durbin-Watson Statistic = 2.0280

Normality Test: Passed (P = 0.7009)

Constant Variance Test: Passed (P = 0.9477)

Power of performed test with alpha = 0.0500: 0.7324

The power of the performed test (0.7324) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0138	-0.0138	-0.5813	-0.7352	-0.6963
2	1.0099	0.0168	0.7071	0.8204	0.7888
3	1.0059	-0.0073	-0.3069	-0.3128	-0.2825
4	1.0020	-0.0197	-0.8291	-0.9171	-0.8994
5	0.9980	0.0169	0.7098	0.6890	0.6477
6	0.9903	0.0297	1.2469	1.3606	1.5335
7	0.9825	0.0007	0.0294	0.0341	0.0305
8	0.9748	-0.0271	-1.1389	-1.3111	-1.4476
9	0.9374	0.0037	0.1555	0.7867	0.7517

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.0811	0.3750	-0.5394	
2	0.0582	0.2571	0.4641	
3	0.0009	0.0374	-0.0557	
4	0.0470	0.1826	-0.4250	
5	-0.0069	-0.0613	(+inf)	
6	0.0883	0.1602	0.6697	
7	0.0001	0.2569	0.0179	
8	0.1398	0.2455	-0.8257	
9	3.8057	0.9609	3.7281	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0138	0.9764	1.0513	0.9421	1.0856
2	1.0099	0.9788	1.0409	0.9413	1.0784
3	1.0059	0.9941	1.0177	0.9436	1.0682
4	1.0020	0.9758	1.0281	0.9355	1.0685
5	0.9980	0.9980	0.9980	0.9980	0.9980
6	0.9903	0.9658	1.0147	0.9244	1.0561
7	0.9825	0.9515	1.0135	0.9139	1.0511
8	0.9748	0.9445	1.0052	0.9066	1.0431
9	0.9374	0.8774	0.9973	0.8517	1.0230

2D Graph 37

.....

TALC (BiPhasic First-order reaction)

40°C / 75%RH

NO OUTPUT

.....

TALC (BiPhasic First-order reaction)

50°C / 20%moisture

Nonlinear Regression

[Variables]

$x = \text{col}(1)$

$y = \text{col}(2)$

$\text{reciprocal_y} = 1/\text{abs}(y)$

$\text{reciprocal_ysquare} = 1/y^2$

'Automatic Initial Parameter Estimate Functions

$\text{xnear0}(q) = \max(\text{abs}(q)) - \text{abs}(q)$

$\text{yatznear0}(q,r) = \text{xatymax}(q, \text{xnear0}(r))$

[Parameters]

$a = \text{yatznear0}(y,x)/2$ "Auto {{previous: 0.341123}}

$b = -\ln(.5)/(0.5*(x50(x,y)-\min(x)))$ "Auto {{previous: 0.516044}}

$c = \text{yatznear0}(y,x)/2$ "Auto {{previous: 0.66249}}

$d = -\ln(.5)/(1.5*(x50(x,y)-\min(x)))$ "Auto {{previous: 0.00541061}}

[Equation]

$f = a * \exp(-b*x) + c * \exp(-d*x)$

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0
d>0
[Options]
tolerance=1e-6
stepsize=0.1
iterations=100

R = 0.9666 Rsqr = 0.9342 Adj Rsqr = 0.8948

Standard Error of Estimate = 0.0441

	Coefficient		Std. Error	t	P
a	0.3411	0.0746	4.5705	0.0060	
b	0.5160	0.2261	2.2820	0.0714	
c	0.6625	0.0694	9.5400	0.0002	
d	0.0054	0.0078	0.6896	0.5211	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.1380	0.0460	23.6729	0.0022
Residual	5	0.0097	0.0019		
Total	8	0.1477	0.0185		

PRESS = 0.0343

Durbin-Watson Statistic = 3.4066

Normality Test: Passed (P = 0.7948)

Constant Variance Test: Passed (P = 0.4342)

Power of performed test with alpha = 0.0500: 0.9988

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0036	-0.0036	-0.0819	-0.2906	-0.2621
2	0.8625	0.0094	0.2126	0.2673	0.2408
3	0.7769	0.0142	0.3224	0.4034	0.3668
4	0.7244	-0.0579	-1.3138	-1.5306	-1.8780
5	0.6916	0.0433	0.9823	1.1077	1.1405
6	0.6568	-0.0167	-0.3785	-0.4448	-0.4060
7	0.6399	0.0473	1.0729	1.3067	1.4403
8	0.6296	-0.0406	-0.9208	-1.1104	-1.1443
9	0.5946	0.0046	0.1050	0.5247	0.4828

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.2443	0.9205	-0.8917	
2	0.0104	0.3673	0.1835	
3	0.0230	0.3612	0.2758	
4	0.2093	0.2632	-1.1226	
5	0.0833	0.2136	0.5944	
6	0.0188	0.2759	-0.2506	
7	0.2063	0.3259	1.0013	
8	0.1401	0.3125	-0.7714	
9	1.6491	0.9599	2.3632	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0036	0.8949	1.1123	0.8466	1.1607
2	0.8625	0.7938	0.9312	0.7300	0.9950
3	0.7769	0.7088	0.8450	0.6447	0.9091
4	0.7244	0.6662	0.7825	0.5970	0.8517
5	0.6916	0.6392	0.7440	0.5668	0.8164
6	0.6568	0.5972	0.7163	0.5287	0.7848
7	0.6399	0.5752	0.7046	0.5094	0.7704
8	0.6296	0.5662	0.6929	0.4997	0.7594
9	0.5946	0.4835	0.7056	0.4359	0.7532

2D Graph 39

TALC (BiPhasic First-order reaction)**50°C**

Nonlinear Regression

[Variables]

 $x = \text{col}(1)$ $y = \text{col}(2)$ $\text{reciprocal_y} = 1/\text{abs}(y)$ $\text{reciprocal_ysquare} = 1/y^2$

'Automatic Initial Parameter Estimate Functions

 $\text{xnear0}(q) = \max(\text{abs}(q)) - \text{abs}(q)$ $\text{yatznear0}(q,r) = \text{xatymax}(q, \text{xnear0}(r))$

[Parameters]

 $a = \text{yatznear0}(y,x)/2$ "Auto {{previous: 0.194689}} $b = -\ln(.5)/(0.5*(x50(x,y) - \min(x)))$ "Auto {{previous: 0.296141}} $c = \text{yatznear0}(y,x)/2$ "Auto {{previous: 0.83071}} $d = -\ln(.5)/(1.5*(x50(x,y) - \min(x)))$ "Auto {{previous: 0.0366477}}

[Equation]

 $f = a * \exp(-b * x) + c * \exp(-d * x)$

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

 $b > 0$ $d > 0$

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

 $R = 0.99183104$ $\text{Rsqr} = 0.98372882$ $\text{Adj Rsqr} = 0.97396611$

Standard Error of Estimate = 0.0316

	Coefficient		Std. Error	t	P
a	0.1947	0.1710	1.1384	0.3065	
b	0.2961	0.3061	0.9675	0.3777	
c	0.8307	0.1778	4.6722	0.0055	
d	0.0366	0.0129	2.8352	0.0365	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.3023	0.1008	100.7639	<0.0001
Residual	5	0.0050	0.0010		
Total	8	0.3073	0.0384		

PRESS = 0.0905

Durbin-Watson Statistic = 2.5529

Normality Test: Passed (P = 0.4865)

Constant Variance Test: Passed (P = 0.0988)

Power of performed test with alpha = 0.0500: 1.0000

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0254	-0.0254	-0.8032	-2.0608	-4.7497
2	0.9456	0.0558	1.7659	2.0983	5.4311
3	0.8797	-0.0077	-0.2440	-0.2935	-0.2648
4	0.8243	-0.0282	-0.8912	-1.0622	-1.0796
5	0.7770	0.0009	0.0274	0.0316	0.0282
6	0.6997	-0.0108	-0.3426	-0.3951	-0.3591
7	0.6378	0.0061	0.1938	0.2412	0.2170
8	0.5859	0.0142	0.4480	0.5911	0.5482
9	0.3997	-0.0051	-0.1625	-1.0915	-1.1186

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	5.9275	0.8481	-11.2226	
2	0.4534	0.2918	3.4858	
3	0.0096	0.3091	-0.1771	
4	0.1186	0.2961	-0.7001	
5	0.0001	0.2485	0.0162	
6	0.0129	0.2481	-0.2063	
7	0.0080	0.3548	0.1609	
8	0.0648	0.4258	0.4721	
9	13.1347	0.9778	-7.4283	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0254	0.9505	1.1003	0.9149	1.1359
2	0.9456	0.9017	0.9895	0.8532	1.0380
3	0.8797	0.8345	0.9249	0.7867	0.9727
4	0.8243	0.7801	0.8685	0.7318	0.9168
5	0.7770	0.7365	0.8175	0.6862	0.8678
6	0.6997	0.6592	0.7402	0.6089	0.7905
7	0.6378	0.5894	0.6862	0.5432	0.7324
8	0.5859	0.5329	0.6389	0.4888	0.6830
9	0.3997	0.3193	0.4800	0.2854	0.5140

2D Graph 40

FERRIC-OXIDE (First-order reaction)

25°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y = 1/abs(y)

reciprocal_ysquare = 1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q) = max(abs(q)) - abs(q)

yatxnear0(q,r) = xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 95.3049}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.00734397}}

[Equation]

f = a * exp(-b * x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b > 0

[Options]

tolerance = 0.0001

stepsize = 100

iterations = 100

R = 0.73300110

Rsqr = 0.53729062

Adj Rsqr = 0.47118928

Standard Error of Estimate = 3.9417

	Coefficient		Std. Error	t	P
a	95.3049	1.9431	49.0490	<0.0001	
b	0.0073	0.0026	2.8097	0.0262	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	126.2918	126.2918	8.1283	0.0247
Residual	7	108.7613	15.5373		
Total	8	235.0531	29.3816		

PRESS = 326.1659

Durbin-Watson Statistic = 2.2618

Normality Test: Passed (P = 0.2642)

Constant Variance Test: Passed (P = 0.4072)

Power of performed test with alpha = 0.0500: 0.6296

The power of the performed test (0.6296) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	95.3049	4.6951	1.1911	1.3690	1.4812
2	94.6075	-4.5163	-1.1458	-1.2809	-1.3553
3	93.9153	3.5307	0.8957	0.9807	0.9775
4	93.2281	0.5700	0.1446	0.1559	0.1446
5	92.5459	0.5543	0.1406	0.1501	0.1392
6	91.1966	0.0533	0.0135	0.0143	0.0133
7	89.8669	-4.3953	-1.1151	-1.1949	-1.2399
8	88.5565	-4.3463	-1.1026	-1.2127	-1.2633
9	82.2861	3.8744	0.9829	1.8391	2.3684

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	0.3008	0.2430	0.8392
2	0.2049	0.1999	-0.6774
3	0.0955	0.1657	0.4357
4	0.0020	0.1402	0.0584
5	0.0016	0.1228	0.0521
6	0.0000	0.1115	0.0047

7	0.1059	0.1292	-0.4776
8	0.1542	0.1733	-0.5785
9	4.2291	0.7143	3.7453

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	95.3049	90.7103	99.8995	84.9132	105.6965
2	94.6075	90.4404	98.7747	84.3977	104.8174
3	93.9153	90.1208	97.7097	83.8518	103.9788
4	93.2281	89.7386	96.7176	83.2756	103.1806
5	92.5459	89.2793	95.8126	82.6693	102.4225
6	91.1966	88.0838	94.3093	81.3698	101.0233
7	89.8669	86.5167	93.2170	79.9623	99.7714
8	88.5565	84.6759	92.4371	78.4603	98.6528
9	82.2861	74.4083	90.1638	70.0821	94.4900

2D Graph 37

FERRIC-OXIDE (First-order reaction)

25°C / 60 %RH

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatknear0(y,x) "Auto {{previous: 94.6625}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.00327568}}

[Equation]

f=a*exp(-b*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

[Options]

tolerance=0.0001

stepsize=100

iterations=100

R = 0.49034996 Rsqr = 0.24044308 Adj Rsqr = 0.13193495

Standard Error of Estimate = 3.4837

	Coefficient		Std. Error	t	P
a	94.6625	1.6889	56.0487	<0.0001	
b	0.0033	0.0022	1.4886	0.1802	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	26.8925	26.8925	2.2159	0.1802
Residual	7	84.9533	12.1362		
Total	8	111.8458	13.9807		

PRESS = 245.4569

Durbin-Watson Statistic = 1.9811

Normality Test: Passed (P = 0.3652)

Constant Variance Test: Passed (P = 0.9129)

Power of performed test with alpha = 0.0500: 0.2592

The power of the performed test (0.2592) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	94.6625	5.3375	1.5321	1.7518	2.1641
2	94.3530	-1.1887	-0.3412	-0.3805	-0.3560

3	94.0444	2.8383	0.8147	0.8913	0.8764
4	93.7368	-2.1949	-0.6300	-0.6796	-0.6510
5	93.4303	-3.4791	-0.9987	-1.0668	-1.0792
6	92.8202	1.5619	0.4483	0.4756	0.4476
7	92.2141	-3.5891	-1.0302	-1.1022	-1.1224
8	91.6119	-2.3332	-0.6697	-0.7341	-0.7074
9	88.6596	3.0512	0.8758	1.7026	2.0594

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.4714	0.2350	1.1996	
2	0.0176	0.1960	-0.1758	
3	0.0782	0.1645	0.3889	
4	0.0377	0.1404	-0.2631	
5	0.0802	0.1236	-0.4052	
6	0.0141	0.1112	0.1583	
7	0.0878	0.1263	-0.4267	
8	0.0543	0.1677	-0.3176	
9	4.0280	0.7354	3.4331	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	94.6625	90.6688	98.6562	85.5078	103.8172
2	94.3530	90.7062	97.9998	85.3442	103.3617
3	94.0444	90.7035	97.3853	85.1551	102.9337
4	93.7368	90.6503	96.8234	84.9399	102.5337
5	93.4303	90.5346	96.3260	84.6985	102.1621
6	92.8202	90.0733	95.5671	84.1366	101.5038
7	92.2141	89.2870	95.1412	83.4719	100.9563
8	91.6119	88.2385	94.9854	82.7103	100.5136
9	88.6596	81.5955	95.7238	77.8079	99.5114

2D Graph 38

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FERRIC-OXIDE (First-order reaction)

40°C / 75 %RH

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 97.0328}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.00384635}}

[Equation]

f=a*exp(-b*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

[Options]

tolerance=0.0001

stepsize=100

iterations=100

R = 0.52010303 Rsqr = 0.27050716 Adj Rsqr = 0.16629390

Standard Error of Estimate = 3.8442

	Coefficient		Std. Error	t	P
a	97.0328	1.8682	51.9404	<0.0001	
b	0.0038	0.0024	1.6110	0.1512	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	38.3596	38.3596	2.5957	0.1512
Residual	7	103.4468	14.7781		
Total	8	141.8064	17.7258		

PRESS = 380.7332

Durbin-Watson Statistic = 1.4961

Normality Test: Passed ($P = 0.2819$)

Constant Variance Test: Passed ($P = 0.1243$)

Power of performed test with $\alpha = 0.0500$: 0.2919

The power of the performed test (0.2919) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	97.0328	2.9672	0.7719	0.8832	0.8674
2	96.6603	-2.0478	-0.5327	-0.5943	-0.5646
3	96.2892	1.4408	0.3748	0.4101	0.3843
4	95.9196	3.2910	0.8561	0.9233	0.9122
5	95.5514	1.8261	0.4750	0.5074	0.4786
6	94.8191	-1.4809	-0.3852	-0.4086	-0.3829
7	94.0925	-5.4011	-1.4050	-1.5034	-1.6915
8	93.3715	-4.9135	-1.2782	-1.4017	-1.5301
9	89.8483	4.3244	1.1249	2.1748	3.5354

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.1206	0.2362	0.4823	
2	0.0432	0.1965	-0.2793	
3	0.0166	0.1647	0.1706	
4	0.0696	0.1404	0.3686	
5	0.0181	0.1235	0.1796	
6	0.0104	0.1112	-0.1355	
7	0.1639	0.1267	-0.6441	
8	0.1991	0.1685	-0.6887	
9	6.4738	0.7324	5.8495	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	97.0328	92.6153	101.4503	86.9261	107.1395
2	96.6603	92.6304	100.6902	86.7169	106.6037
3	96.2892	92.6006	99.9779	86.4792	106.0993
4	95.9196	92.5140	99.3252	86.2124	105.6268
5	95.5514	92.3573	98.7454	85.9164	105.1863
6	94.8191	91.7875	97.8507	85.2368	104.4015
7	94.0925	90.8575	97.3275	84.4439	103.7412
8	93.3715	89.6402	97.1027	83.5453	103.1976
9	89.8483	82.0686	97.6279	77.8836	101.8130

2D Graph 39

FERRIC-OXIDE (First-order reaction)

50°C / 20 %moisture

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 92.2681}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.0060028}}

[Equation]

f=a*exp(-b*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

[Options]

tolerance=0.0001

stepsize=100

iterations=100

R = 0.54301341

Rsqr = 0.29486356

Adj Rsqr = 0.19412978

Standard Error of Estimate = 5.2637

	Coefficient		Std. Error	t	P
a	92.2681	2.5803	35.7592	<0.0001	
b	0.0060	0.0035	1.6961	0.1337	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	81.1015	81.1015	2.9272	0.1308
Residual	7	193.9459	27.7066		
Total	8	275.0474	34.3809		

PRESS = 424.2674

Durbin-Watson Statistic = 2.0540

Normality Test: Passed (P = 0.4458)

Constant Variance Test: Passed (P = 0.2230)

Power of performed test with alpha = 0.0500: 0.3193

The power of the performed test (0.3193) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	92.2681	7.7319	1.4689	1.6853	2.0240
2	91.7159	-6.6647	-1.2662	-1.4143	-1.5494
3	91.1669	0.2321	0.0441	0.0483	0.0447
4	90.6213	6.4185	1.2194	1.3151	1.4031
5	90.0790	-1.6415	-0.3119	-0.3330	-0.3108
6	89.0040	-4.7380	-0.9001	-0.9549	-0.9479
7	87.9418	-2.9907	-0.5682	-0.6085	-0.5789
8	86.8924	-1.7186	-0.3265	-0.3587	-0.3352
9	81.8298	3.3844	0.6430	1.2184	1.2709

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	0.4492	0.2403	1.1383
2	0.2478	0.1986	-0.7712
3	0.0002	0.1653	0.0199
4	0.1411	0.1402	0.5667
5	0.0078	0.1231	-0.1164
6	0.0571	0.1114	-0.3356

7	0.0272	0.1282	-0.2219
8	0.0133	0.1714	-0.1524
9	1.9234	0.7215	2.0457

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	92.2681	86.1667	98.3694	78.4064	106.1297
2	91.7159	86.1694	97.2623	78.0893	105.3424
3	91.1669	86.1062	96.2277	77.7308	104.6031
4	90.6213	85.9601	95.2825	77.3305	103.9122
5	90.0790	85.7124	94.4456	76.8886	103.2694
6	89.0040	84.8500	93.1580	75.8824	102.1256
7	87.9418	83.4860	92.3976	74.7216	101.1620
8	86.8924	81.7393	92.0454	73.4211	100.3636
9	81.8298	71.2572	92.4025	65.4989	98.1608

2D Graph 40

FERRIC-OXIDE (First-order reaction)

50°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

```

a = yatznear0(y,x) "Auto {{previous: 93.9272}}
b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.0435405}}
[Equation]
f=a*exp(-b*x)
fit f to y
"fit f to y with weight reciprocal_y
"fit f to y with weight reciprocal_ysquare
[Constraints]
b>0
[Options]
tolerance=0.0001
stepsize=100
iterations=100

```

R = 0.97200797 Rsqr = 0.94479949 Adj Rsqr = 0.93691370

Standard Error of Estimate = 4.2800

	Coefficient		Std. Error	t	P
a	93.9272	2.4277	38.6891	<0.0001	
b	0.0435	0.0046	9.4872	<0.0001	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	2194.7751	2194.7751	119.8104	<0.0001
Residual	7	128.2311	18.3187		
Total	8	2323.0062	290.3758		

PRESS = 287.4305

Durbin-Watson Statistic = 1.4476

Normality Test: Passed (P = 0.7036)

Constant Variance Test: Passed (P = 0.8437)

Power of performed test with alpha = 0.0500: 0.9994

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	93.9272	6.0728	1.4189	1.7228	2.1017
2	89.9254	-2.0340	-0.4752	-0.5423	-0.5130
3	86.0940	1.8044	0.4216	0.4634	0.4357
4	82.4258	1.0546	0.2464	0.2651	0.2467
5	78.9140	-5.2288	-1.2217	-1.3019	-1.3845
6	72.3328	-2.1438	-0.5009	-0.5362	-0.5069

7	66.3004	-4.2779	-0.9995	-1.0971	-1.1162
8	60.7712	-0.0489	-0.0114	-0.0130	-0.0121
9	39.3191	5.7103	1.3342	1.8711	2.4503

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.7040	0.3217	1.4475	
2	0.0444	0.2320	-0.2820	
3	0.0223	0.1721	0.1987	
4	0.0055	0.1362	0.0980	
5	0.1149	0.1194	-0.5098	
6	0.0210	0.1272	-0.1935	
7	0.1232	0.1700	-0.5051	
8	0.0000	0.2297	-0.0066	
9	1.6927	0.4916	2.4095	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	93.9272	88.1865	99.6679	82.2918	105.5627
2	89.9254	85.0501	94.8006	78.6916	101.1591
3	86.0940	81.8952	90.2928	75.1369	97.0511
4	82.4258	78.6909	86.1608	71.6380	93.2137
5	78.9140	75.4171	82.4108	68.2062	89.6217
6	72.3328	68.7228	75.9427	61.5876	83.0780
7	66.3004	62.1282	70.4727	55.3535	77.2474
8	60.7712	55.9201	65.6222	49.5479	71.9944
9	39.3191	32.2231	46.4151	26.9586	51.6796

2D Graph 41

FERRIC-OXIDE (BiPhasic First-order reaction)**25°C**

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatznear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatznear0(y,x)/2 "Auto {{previous: 0.141982}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.164601}}

c = yatznear0(y,x)/2 "Auto {{previous: 0.840774}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 1.87663e-010}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.83352356 Rsqr = 0.69476153 Adj Rsqr = 0.51161844

Standard Error of Estimate = 0.0379

	Coefficient		Std. Error	t	P
a	0.1420	0.4079	0.3480	0.7420	
b	0.1646	0.5184	0.3175	0.7637	
c	0.8408	0.4224	1.9905	0.1032	
d	0.0000	0.0218	0.0000	1.0000	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.0163	0.0054	3.7935	0.0927
Residual	5	0.0072	0.0014		
Total	8	0.0235	0.0029		

PRESS = 19.2427

Durbin-Watson Statistic = 2.5793

Normality Test: Failed (P = 0.0160)

Constant Variance Test: Passed (P = 0.5198)

Power of performed test with alpha = 0.0500: 0.8361

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	0.9828	0.0172	0.4552	0.9542	0.9437
2	0.9612	-0.0603	-1.5917	-1.8642	-3.0196
3	0.9429	0.0315	0.8324	0.9637	0.9552
4	0.9274	0.0106	0.2786	0.3308	0.2991
5	0.9143	0.0167	0.4416	0.5211	0.4792
6	0.8937	0.0188	0.4974	0.5679	0.5252
7	0.8788	-0.0241	-0.6364	-0.7809	-0.7454
8	0.8682	-0.0260	-0.6876	-1.0426	-1.0542
9	0.8461	0.0156	0.4106	6.8933	(+inf)

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.7725	0.7724	1.7385	
2	0.3230	0.2710	-1.8411	
3	0.0790	0.2540	0.5574	
4	0.0112	0.2903	0.1913	
5	0.0266	0.2818	0.3002	
6	0.0245	0.2330	0.2895	
7	0.0771	0.3359	-0.5302	
8	0.3531	0.5650	-1.2015	
9	3337.0459	0.9965	(+inf)	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	0.9828	0.8972	1.0683	0.8531	1.1124
2	0.9612	0.9105	1.0119	0.8514	1.0710
3	0.9429	0.8939	0.9920	0.8339	1.0520
4	0.9274	0.8750	0.9799	0.8168	1.0380
5	0.9143	0.8626	0.9660	0.8040	1.0245
6	0.8937	0.8466	0.9407	0.7855	1.0018
7	0.8788	0.8224	0.9353	0.7663	0.9914
8	0.8682	0.7950	0.9413	0.7463	0.9900
9	0.8461	0.7489	0.9433	0.7085	0.9836

2D Graph 1

FERRIC-OXIDE (BiPhasic First-order reaction)

25°C / 60 %RH

Nonlinear Regression

[Variables]

$x = \text{col}(1)$

$y = \text{col}(2)$

$\text{reciprocal_y} = 1/\text{abs}(y)$

$\text{reciprocal_ysquare} = 1/y^2$

'Automatic Initial Parameter Estimate Functions

$\text{xnear0}(q) = \max(\text{abs}(q)) - \text{abs}(q)$

$\text{yatznear0}(q,r) = \text{xatymax}(q, \text{xnear0}(r))$

[Parameters]

$a = \text{yatznear0}(y,x)/2$ "Auto {{previous: 0.087789}}

$b = -\ln(.5)/(0.5*(x50(x,y) - \min(x)))$ "Auto {{previous: 0.531831}}

$c = \text{yatznear0}(y,x)/2$ "Auto {{previous: 0.905793}}

$d = -\ln(.5)/(1.5*(x50(x,y) - \min(x)))$ "Auto {{previous: 9.84071e-012}}

[Equation]

$f = a * \exp(-b * x) + c * \exp(-d * x)$

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

$b > 0$

$d > 0$

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.79886257 Rsqr = 0.63818141 Adj Rsqr = 0.42109026

Standard Error of Estimate = 0.0284

	Coefficient		Std. Error	t	P
a	0.0878	0.0450	1.9503	0.1086	
b	0.5318	0.5658	0.9400	0.3904	
c	0.9058	0.0407	22.2519	<0.0001	
d	0.0000	0.0033	0.0000	1.0000	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.0071	0.0024	2.9397	0.1380
Residual	5	0.0040	0.0008		
Total	8	0.0112	0.0014		

PRESS = 0.1041

Durbin-Watson Statistic = 3.0961

Normality Test: Passed (P = 0.3683)

Constant Variance Test: Passed (P = 0.9129)

Power of performed test with alpha = 0.0500: 0.7653

The power of the performed test (0.7653) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	0.9936	0.0064	0.2256	0.8197	0.7880
2	0.9574	-0.0257	-0.9044	-1.1430	-1.1895
3	0.9361	0.0327	1.1505	1.4414	1.6864
4	0.9236	-0.0082	-0.2875	-0.3341	-0.3022
5	0.9163	-0.0167	-0.5885	-0.6624	-0.6204
6	0.9094	0.0344	1.2097	1.4209	1.6458
7	0.9070	-0.0208	-0.7308	-0.8881	-0.8656
8	0.9062	-0.0134	-0.4723	-0.5678	-0.5251
9	0.9058	0.0113	0.3976	2.0390	4.4425

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	2.0496	0.9243	2.7526

2	0.1951	0.3740	-0.9194
3	0.2959	0.3630	1.2729
4	0.0098	0.2597	-0.1790
5	0.0293	0.2108	-0.3206
6	0.1915	0.2751	1.0139
7	0.0941	0.3230	-0.5979
8	0.0359	0.3082	-0.3505
9	26.2892	0.9620	22.3428

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	0.9936	0.9233	1.0639	0.8921	1.0950
2	0.9574	0.9126	1.0021	0.8716	1.0431
3	0.9361	0.8920	0.9802	0.8507	1.0215
4	0.9236	0.8863	0.9609	0.8415	1.0057
5	0.9163	0.8827	0.9498	0.8358	0.9967
6	0.9094	0.8710	0.9478	0.8268	0.9920
7	0.9070	0.8655	0.9486	0.8229	0.9912
8	0.9062	0.8656	0.9468	0.8226	0.9899
9	0.9058	0.8341	0.9775	0.8034	1.0082

2D Graph 2

FERRIC-OXIDE (BiPhasic First-order reaction)

40°C / 75 %RH

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

```

reciprocal_y=1/abs(y)
reciprocal_ysquare=1/y^2
'Automatic Initial Parameter Estimate Functions
xnear0(q)=max(abs(q))-abs(q)
yatxnear0(q,r)=xatymax(q,xnear0(r))
[Parameters]
a = yatxnear0(y,x)/2 "Auto {{previous: 0.0903676}}
b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.210639}}
c = yatxnear0(y,x)/2 "Auto {{previous: 0.907939}}
d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 3.32243e-011}}
[Equation]
f=a*exp(-b*x)+c*exp(-d*x)
fit f to y
"fit f to y with weight reciprocal_y
"fit f to y with weight reciprocal_ysquare
[Constraints]
b>0
d>0
[Options]
tolerance=1e-6
stepsize=0.1
iterations=100

```

R = 0.71054287 Rsqr = 0.50487117 Adj Rsqr = 0.20779387

Standard Error of Estimate = 0.0375

	Coefficient		Std. Error	t	P
a	0.0904	0.2259	0.4001	0.7056	
b	0.2106	0.6808	0.3094	0.7695	
c	0.9079	0.2377	3.8203	0.0124	
d	0.0000	0.0128	0.0000	1.0000	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.0072	0.0024	1.6995	0.2816
Residual	5	0.0070	0.0014		
Total	8	0.0142	0.0018		

PRESS = 36.0903

Durbin-Watson Statistic = 1.5338

Normality Test: Passed (P = 0.2873)

Constant Variance Test: Passed (P = 0.5198)

Power of performed test with $\alpha = 0.0500$: 0.5855

The power of the performed test (0.5855) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	0.9983	0.0017	0.0452	0.1000	0.0896
2	0.9811	-0.0350	-0.9345	-1.0962	-1.1249
3	0.9672	0.0101	0.2685	0.3140	0.2836
4	0.9560	0.0361	0.9642	1.1481	1.1967
5	0.9469	0.0269	0.7185	0.8422	0.8131
6	0.9335	-0.0001	-0.0024	-0.0028	-0.0025
7	0.9247	-0.0378	-1.0082	-1.2436	-1.3384
8	0.9189	-0.0344	-0.9168	-1.3336	-1.4860
9	0.9093	0.0324	0.8659	11.7811	(+inf)

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.0098	0.7960	0.1769	
2	0.1129	0.2732	-0.6897	
3	0.0091	0.2687	0.1719	
4	0.1377	0.2948	0.7736	
5	0.0663	0.2722	0.4973	
6	0.0000	0.2303	-0.0014	
7	0.2016	0.3427	-0.9665	
8	0.4962	0.5274	-1.5699	
9	6387.8017	0.9946	(+inf)	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	0.9983	0.9124	1.0842	0.8692	1.1274
2	0.9811	0.9308	1.0315	0.8724	1.0898
3	0.9672	0.9173	1.0172	0.8587	1.0757
4	0.9560	0.9037	1.0083	0.8464	1.0656
5	0.9469	0.8966	0.9971	0.8382	1.0555
6	0.9335	0.8872	0.9797	0.8266	1.0403
7	0.9247	0.8683	0.9811	0.8131	1.0363
8	0.9189	0.8490	0.9889	0.7999	1.0380
9	0.9093	0.8132	1.0053	0.7732	1.0453

2D Graph 3

FERRIC-OXIDE (BiPhasic First-order reaction)

50°C / 20 %moisture

Nonlinear Regression

[Variables]

$x = \text{col}(1)$

$y = \text{col}(2)$

$\text{reciprocal_y} = 1/\text{abs}(y)$

$\text{reciprocal_ysquare} = 1/y^2$

'Automatic Initial Parameter Estimate Functions

$\text{xnear0}(q) = \max(\text{abs}(q)) - \text{abs}(q)$

$\text{yatznear0}(q,r) = \text{xatymax}(q, \text{xnear0}(r))$

[Parameters]

$a = \text{yatznear0}(y,x)/2$ "Auto {{previous: 0.118032}}

$b = -\ln(.5)/(0.5*(x50(x,y) - \min(x)))$ "Auto {{previous: 0.284503}}

$c = \text{yatznear0}(y,x)/2$ "Auto {{previous: 0.845865}}

$d = -\ln(.5)/(1.5*(x50(x,y) - \min(x)))$ "Auto {{previous: 1.2391e-011}}

[Equation]

$f = a * \exp(-b * x) + c * \exp(-d * x)$

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

$b > 0$

$d > 0$

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.68453483 Rsqr = 0.46858793 Adj Rsqr = 0.14974069

Standard Error of Estimate = 0.0541

	Coefficient		Std. Error	t	P
a	0.1180	0.1725	0.6841	0.5243	
b	0.2845	0.6735	0.4224	0.6903	
c	0.8459	0.1825	4.6351	0.0057	
d	0.0000	0.0122	0.0000	1.0000	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.0129	0.0043	1.4696	0.3290
Residual	5	0.0146	0.0029		
Total	8	0.0275	0.0034		

PRESS = 0.4189

Durbin-Watson Statistic = 2.2924

Normality Test: Passed (P = 0.2163)

Constant Variance Test: Passed (P = 0.1384)

Power of performed test with alpha = 0.0500: 0.5365

The power of the performed test (0.5365) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	0.9639	0.0361	0.6677	1.6347	2.1430
2	0.9347	-0.0842	-1.5566	-1.8376	-2.8849
3	0.9127	0.0013	0.0242	0.0288	0.0258
4	0.8961	0.0743	1.3735	1.6375	2.1509
5	0.8837	0.0007	0.0127	0.0147	0.0131
6	0.8673	-0.0246	-0.4553	-0.5200	-0.4782
7	0.8580	-0.0085	-0.1567	-0.1944	-0.1745
8	0.8527	-0.0010	-0.0183	-0.0250	-0.0224
9	0.8463	0.0059	0.1087	1.0876	1.1133

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	3.3361	0.8332	4.7888

2	0.3324	0.2825	-1.8103
3	0.0001	0.2952	0.0167
4	0.2825	0.2965	1.3963
5	0.0000	0.2537	0.0077
6	0.0206	0.2334	-0.2639
7	0.0051	0.3501	-0.1281
8	0.0001	0.4655	-0.0209
9	29.2924	0.9900	11.0807

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	0.9639	0.8370	1.0908	0.7757	1.1521
2	0.9347	0.8608	1.0085	0.7773	1.0921
3	0.9127	0.8372	0.9882	0.7545	1.0709
4	0.8961	0.8205	0.9718	0.7379	1.0544
5	0.8837	0.8137	0.9537	0.7281	1.0393
6	0.8673	0.8001	0.9344	0.7129	1.0216
7	0.8580	0.7758	0.9402	0.6965	1.0195
8	0.8527	0.7579	0.9476	0.6845	1.0210
9	0.8463	0.7080	0.9846	0.6502	1.0423

2D Graph 4

FERRIC-OXIDE (BiPhasic First-order reaction)

50°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

```

reciprocal_y=1/abs(y)
reciprocal_ysquare=1/y^2
'Automatic Initial Parameter Estimate Functions
xnear0(q)=max(abs(q))-abs(q)
yatxnear0(q,r)=xatymax(q,xnear0(r))
[Parameters]
a = yatxnear0(y,x)/2 "Auto {{previous: 0.278258}}
b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.21828}}
c = yatxnear0(y,x)/2 "Auto {{previous: 0.709415}}
d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 0.0230322}}
[Equation]
f=a*exp(-b*x)+c*exp(-d*x)
fit f to y
"fit f to y with weight reciprocal_y
"fit f to y with weight reciprocal_ysquare
[Constraints]
b>0
d>0
[Options]
tolerance=1e-6
stepsize=0.1
iterations=100

```

R = 0.99107522 Rsqr = 0.98223009 Adj Rsqr = 0.97156814

Standard Error of Estimate = 0.0287

	Coefficient		Std. Error	t	P
a	0.2783	0.2471	1.1260	0.3113	
b	0.2183	0.2021	1.0802	0.3294	
c	0.7094	0.2563	2.7681	0.0395	
d	0.0230	0.0178	1.2934	0.2524	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.2282	0.0761	92.1248	<0.0001
Residual	5	0.0041	0.0008		
Total	8	0.2323	0.0290		

PRESS = 0.0203

Durbin-Watson Statistic = 2.9617

Normality Test: Passed (P = 0.2977)

Constant Variance Test: Passed (P = 0.1384)

Power of performed test with $\alpha = 0.0500$: 1.0000

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	0.9877	0.0123	0.4290	0.9777	0.9724
2	0.9170	-0.0380	-1.3239	-1.5562	-1.9382
3	0.8573	0.0217	0.7546	0.8879	0.8653
4	0.8066	0.0282	0.9811	1.1699	1.2279
5	0.7632	-0.0263	-0.9167	-1.0712	-1.0915
6	0.6930	0.0089	0.3109	0.3553	0.3219
7	0.6386	-0.0183	-0.6385	-0.7911	-0.7565
8	0.5948	0.0124	0.4310	0.6098	0.5669
9	0.4511	-0.0008	-0.0277	-0.2942	-0.2654

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	1.0023	0.8075	1.9914	
2	0.2310	0.2762	-1.1973	
3	0.0758	0.2778	0.5366	
4	0.1444	0.2967	0.7976	
5	0.1048	0.2676	-0.6597	
6	0.0096	0.2340	0.1779	
7	0.0837	0.3485	-0.5532	
8	0.0932	0.5006	0.5676	
9	2.4198	0.9911	-2.8071	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	0.9877	0.9213	1.0540	0.8884	1.0870
2	0.9170	0.8781	0.9558	0.8335	1.0004
3	0.8573	0.8184	0.8962	0.7738	0.9408
4	0.8066	0.7664	0.8468	0.7225	0.8907
5	0.7632	0.7250	0.8014	0.6800	0.8463
6	0.6930	0.6572	0.7287	0.6109	0.7750
7	0.6386	0.5950	0.6822	0.5528	0.7243
8	0.5948	0.5426	0.6471	0.5044	0.6853
9	0.4511	0.3776	0.5246	0.3469	0.5553

2D Graph 5

L-4 Tablets (First-order reaction)

25°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y = 1/abs(y)

reciprocal_ysquare = 1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q) = max(abs(q)) - abs(q)

yatxnear0(q,r) = xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 88.5058}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.0151329}}

[Equation]

f = a * exp(-b * x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b > 0

[Options]

tolerance = 0.0001

stepsize = 100

iterations = 100

R = 0.53591830

Rsqr = 0.28720842

Adj Rsqr = 0.04961123

Standard Error of Estimate = 9.5871

	Coefficient		Std. Error	t	P
a	88.5058	6.2926	14.0651	0.0008	
b	0.0151	0.0138	1.0965	0.3530	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	111.1047	111.1047	1.2088	0.3519
Residual	3	275.7389	91.9130		
Total	4	386.8436	96.7109		

PRESS = 684.7905

Durbin-Watson Statistic = 1.6705

Normality Test: Passed (P = 0.5885)

Constant Variance Test: Passed (P = 0.0500)

Power of performed test with alpha = 0.0500: 0.1327

The power of the performed test (0.1327) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	88.5058	11.4942	1.1989	1.5891	3.2620
2	87.1765	-3.9248	-0.4094	-0.4970	-0.4236
3	85.8672	-11.0416	-1.1517	-1.3287	-1.6912
4	79.6098	2.0661	0.2155	0.2645	0.2185
5	76.0764	1.4259	0.1487	0.2561	0.2115

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	0.9557	0.4308	2.8379
2	0.0585	0.3214	-0.2915
3	0.2921	0.2487	-0.9729
4	0.0177	0.3363	0.1556
5	0.0645	0.6628	0.2965

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	88.5058	68.4800	108.5316	52.0104	125.0012
2	87.1765	69.8791	104.4740	52.1039	122.2491
3	85.8672	70.6529	101.0816	51.7738	119.9606

4	79.6098	61.9173	97.3024	44.3407	114.8790
5	76.0764	51.2364	100.9165	36.7329	115.4200

L-4 Tablets (First-order reaction)

25°C / 60 % RH

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 90.9752}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.00855306}}

[Equation]

f=a*exp(-b*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

[Options]

tolerance=0.0001

stepsize=100

iterations=100

R = 0.36269390 Rsqr = 0.13154686 Adj Rsqr = 0.00000000

Standard Error of Estimate = 9.5249

	Coefficient		Std. Error	t	P
a	90.9752	6.2061	14.6590	0.0007	
b	0.0086	0.0128	0.6674	0.5523	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	41.2269	41.2269	0.4544	0.5485
Residual	3	272.1737	90.7246		
Total	4	313.4006	78.3502		

PRESS = 768.9416

Durbin-Watson Statistic = 2.4655

Normality Test: Passed (P = 0.1734)

Constant Variance Test: Passed (P = 0.0500)

Power of performed test with alpha = 0.0500: 0.0774

The power of the performed test (0.0774) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	90.9752	9.0248	0.9475	1.2490	1.4720
2	90.2004	-6.7429	-0.7079	-0.8595	-0.8083
3	89.4322	-7.0152	-0.7365	-0.8510	-0.7977
4	85.6883	8.8798	0.9323	1.1388	1.2341
5	83.5175	-4.1468	-0.4354	-0.7615	-0.6922

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.5754	0.4245	1.2643	
2	0.1751	0.3216	-0.5565	
3	0.1213	0.2509	-0.4617	
4	0.3192	0.3299	0.8659	
5	0.5970	0.6731	-0.9933	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	90.9752	71.2248	110.7257	54.7961	127.1544
2	90.2004	73.0112	107.3896	55.3534	125.0475
3	89.4322	74.2480	104.6164	55.5293	123.3352
4	85.6883	68.2786	103.0979	50.7319	120.6446
5	83.5175	58.6480	108.3870	44.3086	122.7265

..... L-4 Tablets (First-order reaction)

40°C / 75 % RH

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 93.395}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.0231032}}

[Equation]

f=a*exp(-b*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

[Options]

tolerance=0.0001

stepsize=100

iterations=100

R = 0.77076136 Rsqr = 0.59407308 Adj Rsqr = 0.45876410

Standard Error of Estimate = 7.7486

	Coefficient		Std. Error	t	P
a	93.3950	5.1325	18.1968	0.0004	
b	0.0231	0.0111	2.0775	0.1293	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	263.6055	263.6055	4.3905	0.1271
Residual	3	180.1202	60.0401		
Total	4	443.7258	110.9314		

PRESS = 697.1267

Durbin-Watson Statistic = 1.5207

Normality Test: Passed (P = 0.4144)

Constant Variance Test: Passed (P = 0.0500)

Power of performed test with alpha = 0.0500: 0.3035

The power of the performed test (0.3035) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	93.3950	6.6050	0.8524	1.1378	1.2322
2	91.2621	0.1846	0.0238	0.0289	0.0236
3	89.1778	-6.5428	-0.8444	-0.9724	-0.9594
4	79.4491	-6.9326	-0.8947	-1.1051	-1.1718
5	74.1290	6.7522	0.8714	1.4722	2.2816

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	0.5060	0.4388	1.0894	
2	0.0002	0.3211	0.0162	
3	0.1542	0.2460	-0.5480	

4	0.3210	0.3445	-0.8496
5	2.0093	0.6496	3.1068

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	93.3950	77.0612	109.7289	63.8168	122.9733
2	91.2621	77.2884	105.2357	62.9188	119.6053
3	89.1778	76.9482	101.4074	61.6525	116.7031
4	79.4491	64.9746	93.9235	50.8555	108.0426
5	74.1290	54.2537	94.0043	42.4571	105.8009

2D Graph 48

..... **L-4 Tablets (First-order reaction)**

50°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x) "Auto {{previous: 88.9987}}

b = -ln(.5)/(x50(x,y)-min(x)) "Auto {{previous: 0.0633019}}

[Equation]

f=a*exp(-b*x)

fit f to y
 "fit f to y with weight reciprocal_y
 "fit f to y with weight reciprocal_ysquare
 [Constraints]
 b>0
 [Options]
 tolerance=0.0001
 stepsize=100
 iterations=100

R = 0.90949854 Rsqr = 0.82718759 Adj Rsqr = 0.76958346

Standard Error of Estimate = 9.2619

	Coefficient		Std. Error	t	P
a	88.9987	6.4103	13.8836	0.0008	
b	0.0633	0.0182	3.4759	0.0402	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	1	1231.8276	1231.8276	14.3599	0.0322
Residual	3	257.3480	85.7827		
Total	4	1489.1757	372.2939		

PRESS = 821.6253

Durbin-Watson Statistic = 2.0680

Normality Test: Passed (P = 0.4730)

Constant Variance Test: Passed (P = 0.0500)

Power of performed test with alpha = 0.0500: 0.5778

The power of the performed test (0.5778) is below the desired power of 0.8000.
 You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	88.9987	11.0013	1.1878	1.6456	4.3080
2	83.5396	-9.8140	-1.0596	-1.2831	-1.5596
3	78.4152	-3.1642	-0.3416	-0.3909	-0.3276
4	57.1402	-2.4821	-0.2680	-0.3423	-0.2851
5	47.2571	4.8819	0.5271	0.8133	0.7521

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	1.2451	0.4790	4.1309	
2	0.3838	0.3180	-1.0650	
3	0.0236	0.2360	-0.1821	
4	0.0370	0.3870	-0.2265	
5	0.4566	0.5799	0.8837	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	88.9987	68.5982	109.3992	53.1521	124.8453
2	83.5396	66.9176	100.1615	49.7004	117.3787
3	78.4152	64.0957	92.7347	45.6457	111.1848
4	57.1402	38.8039	75.4766	22.4268	91.8536
5	47.2571	24.8103	69.7038	10.2077	84.3064

L-4 Tablets (BiPhasic First-order reaction)

25°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

```

a = yatxnear0(y,x)/2 "Auto {{previous: 0.219076}}
b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 1.7807}}
c = yatxnear0(y,x)/2 "Auto {{previous: 0.782068}}
d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 7.60564e-011}}

```

[Equation]

```
f=a*exp(-b*x)+c*exp(-d*x)
```

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.95990266 Rsqr = 0.92141311 Adj Rsqr = 0.68565244

Standard Error of Estimate = 0.0551

	Coefficient		Std. Error	t	P
a	0.2191	0.1165	1.8812	0.3110	
b	1.7807	2.8185	0.6318	0.6413	
c	0.7821	0.1039	7.5271	0.0841	
d	0.0000	0.0168	0.0000	1.0000	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.0356	0.0119	3.9083	0.3522
Residual	1	0.0030	0.0030		
Total	4	0.0387	0.0097		

PRESS = 55.5621

Durbin-Watson Statistic = 3.4242

Normality Test: Passed (P = 0.6010)

Constant Variance Test: Passed (P = 0.0500)

Power of performed test with alpha = 0.0500: 0.7853

The power of the performed test (0.7853) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0011	-0.0011	-0.0207	-1.6711	(+inf)
2	0.8190	0.0135	0.2454	1.6706	(+inf)
3	0.7883	-0.0400	-0.7261	-1.6699	(+inf)
4	0.7821	0.0347	0.6292	0.8277	0.0000
5	0.7821	-0.0070	-0.1278	-0.2779	0.0000

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	4532.0189	0.9998	(+inf)
2	31.6346	0.9784	(+inf)
3	2.9906	0.8110	(+inf)
4	0.1252	0.4222	0.0000
5	0.0720	0.7886	0.0000

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0011	0.3006	1.7017	0.0104	1.9919
2	0.8190	0.1260	1.5120	-0.1664	1.8044
3	0.7883	0.1574	1.4192	-0.1545	1.7311
4	0.7821	0.3268	1.2373	-0.0534	1.6176
5	0.7821	0.1599	1.4042	-0.1549	1.7190

2D Graph 9

L-4 Tablets (BiPhasic First-order reaction)**25°C / 60 %RH**

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.408332}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.00882357}}

c = yatxnear0(y,x)/2 "Auto {{previous: 0.501416}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 0.00832282}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.36269364 Rsqr = 0.13154668 Adj Rsqr = 0.00000000

Standard Error of Estimate = 0.1650

	Coefficient		Std. Error	t	P
a	0.4083	6871896.7094	0.0000	1.0000	
b	0.0088	14244.6038	0.0000	1.0000	
c	0.5014	6871896.6285	0.0000	1.0000	
d	0.0083	11689.2197	0.0000	1.0000	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.0041	0.0014	0.0505	0.9789
Residual	1	0.0272	0.0272		
Total	4	0.0313	0.0078		

PRESS = 0.7549

Durbin-Watson Statistic = 2.4654

Normality Test: Passed (P = 0.1730)

Constant Variance Test: Passed (P = 0.0500)

Power of performed test with alpha = 0.0500: 0.0774

The power of the performed test (0.0774) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	0.9097	0.0903	0.5471	0.7737	0.0000
2	0.9020	-0.0674	-0.4087	-1.4294	(+inf)
3	0.8943	-0.0702	-0.4253	-0.6540	0.0000
4	0.8569	0.0888	0.5381	0.5649	0.0000
5	0.8352	-0.0415	-0.2516	-0.3298	0.0000

Influence Diagnostics:

Row	Cook'sDist	Leverage	DFFITS
1	-0.4489	1.5000	(+inf)
2	-6.7584	1.0818	(+inf)
3	-0.3598	1.4229	(+inf)
4	-0.1677	1.9072	(+inf)
5	-0.0739	1.5822	(+inf)

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	0.9097	-1.6576	3.4771	-2.4047	4.2242
2	0.9020	-1.2782	3.0822	-2.1225	3.9265
3	0.8943	-1.6061	3.3948	-2.3686	4.1572
4	0.8569	-2.0380	3.7518	-2.7173	4.4311
5	0.8352	-1.8015	3.4719	-2.5332	4.2037

2D Graph 10

L-4 Tablets (BiPhasic First-order reaction)

40°C / 75%RH

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.24046}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 0.606988}}

c = yatxnear0(y,x)/2 "Auto {{previous: 0.765575}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 4.32791e-010}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y

"fit f to y with weight reciprocal_y

"fit f to y with weight reciprocal_ysquare

[Constraints]

b>0

d>0

[Options]

tolerance=1e-6

stepsize=0.1

iterations=100

R = 0.95135409 Rsqr = 0.90507461 Adj Rsqr = 0.62029845

Standard Error of Estimate = 0.0649

	Coefficient		Std. Error	t	P
a	0.2405	0.3286	0.7317	0.5978	
b	0.6070	1.1249	0.5396	0.6850	
c	0.7656	0.3293	2.3246	0.2586	
d	0.0000	0.0472	0.0000	1.0000	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.0402	0.0134	3.1782	0.3860
Residual	1	0.0042	0.0042		
Total	4	0.0444	0.0111		

PRESS = 2.7170

Durbin-Watson Statistic = 2.3671

Normality Test: Passed (P = 0.6964)

Constant Variance Test: Passed (P = 0.0500)

Power of performed test with alpha = 0.0500: 0.7423

The power of the performed test (0.7423) is below the desired power of 0.8000.
You should interpret the negative findings cautiously.

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0060	-0.0060	-0.0930	-0.5566	0.0000
2	0.8966	0.0178	0.2749	0.4228	0.0000
3	0.8370	-0.0106	-0.1640	-0.2543	0.0000
4	0.7690	-0.0438	-0.6756	-2.0714	(+inf)
5	0.7661	0.0427	0.6576	4.0019	(+inf)

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	2.6970	0.9721	0.0000	
2	0.0610	0.5772	0.0000	
3	0.0227	0.5841	0.0000	
4	9.0114	0.8936	(+inf)	
5	144.2554	0.9730	(+inf)	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0060	0.1930	1.8191	-0.1520	2.1641
2	0.8966	0.2701	1.5232	-0.1390	1.9323
3	0.8370	0.2068	1.4672	-0.2009	1.8749
4	0.7690	-0.0105	1.5486	-0.3658	1.9038
5	0.7661	-0.0473	1.5796	-0.3922	1.9244

L-4 Tablets (BiPhasic First-order reaction)

50°C

Nonlinear Regression

[Variables]

x = col(1)

y = col(2)

reciprocal_y=1/abs(y)

reciprocal_ysquare=1/y^2

'Automatic Initial Parameter Estimate Functions

xnear0(q)=max(abs(q))-abs(q)

yatxnear0(q,r)=xatymax(q,xnear0(r))

[Parameters]

a = yatxnear0(y,x)/2 "Auto {{previous: 0.209693}}

b = -ln(.5)/(0.5*(x50(x,y)-min(x))) "Auto {{previous: 19542.4}}

c = yatxnear0(y,x)/2 "Auto {{previous: 0.790307}}

d = -ln(.5)/(1.5*(x50(x,y)-min(x))) "Auto {{previous: 0.0450895}}

[Equation]

f=a*exp(-b*x)+c*exp(-d*x)

fit f to y
 "fit f to y with weight reciprocal_y
 "fit f to y with weight reciprocal_ysquare
 [Constraints]
 b>0
 d>0
 [Options]
 tolerance=1e-6
 stepsize=0.1
 iterations=100

R = 0.99169616 Rsqr = 0.98346127 Adj Rsqr = 0.93384507

Standard Error of Estimate = 0.0496

	Coefficient		Std. Error	t	P
a	0.2097	0.0676	3.1032	0.1985	
b	19542.4191		2469070.0313	0.0079	0.9950
c	0.7903	0.0459	17.2332	0.0369	
d	0.0451	0.0113	3.9918	0.1563	

Analysis of Variance:

	DF	SS	MS	F	P
Regression	3	0.1465	0.0488	19.8214	0.1633
Residual	1	0.0025	0.0025		
Total	4	0.1489	0.0372		

PRESS = 0.0000

Durbin-Watson Statistic = 3.4872

Normality Test: Passed (P = 0.7092)

Constant Variance Test: Passed (P = 0.0500)

Power of performed test with alpha = 0.0500: 0.9723

Regression Diagnostics:

Row	Predicted	Residual	Std. Res.	Stud. Res.	Stud. Del. Res.
1	1.0000	0.0000	0.0000	(+inf)	(+inf)
2	0.7555	-0.0182	-0.3669	-0.5673	0.0000
3	0.7222	0.0304	0.6116	0.7923	0.0000
4	0.5764	-0.0298	-0.6008	-0.7556	0.0000
5	0.5035	0.0179	0.3611	0.6071	0.0000

Influence Diagnostics:

Row	Cook'sDist		Leverage	DFFITS
1	(+inf)	1.0000	(+inf)	
2	0.1119	0.5817	0.0000	
3	0.1064	0.4041	0.0000	
4	0.0831	0.3679	0.0000	
5	0.1684	0.6463	0.0000	

95% Confidence:

Row	Predicted	Regr. 5%	Regr. 95%	Pop. 5%	Pop. 95%
1	1.0000	0.3694	1.6306	0.1082	1.8918
2	0.7555	0.2745	1.2364	-0.0376	1.5485
3	0.7222	0.3213	1.1230	-0.0250	1.4694
4	0.5764	0.1939	0.9589	-0.1611	1.3139
5	0.5035	-0.0035	1.0104	-0.3056	1.3126

2D Graph 12

APPENDIX B

INTRODUCTION:

This appendix includes the residual plots for the output generated in Appendix A. Using a suitable graphical procedure (Microsoft® Excel 97), residual plots are drawn for the degradation data to fit first order and biphasic first order reactions. This was done for all the drug-excipient mixtures for all conditions. Residuals are plotted against experimentally determined values (observed) and model fitted values (predicted values). Essentially what is considered important while looking at residual plots is a random scatter around the baseline with no significant trend in the plot.

1) Drug + Dextrose

2) Drug + DiCalcium Phosphate Dihydrate

3) Drug + Calcium Sulfate

4) Drug + Mannitol

5) Drug + Lactose Anhydrous

6) Lactose Hydrus

7) Drug + Starch

8) Drug + Talc

9) Ferric Oxide

10) Levothyroxine Sodium Tablets

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