# A CALIBRE SCIENTIFIC COMPANY



## **Titration Standards and Solutions**



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#### **About Reagecon**

Reagecon, part of the Calibre Scientific Group of companies is one of the largest producers of Physical and Chemical Standards. The company is based in an 8,000 sq. metre facility that includes a large suite of manufacturing, quality control and research and development laboratories in Shannon, Ireland with sales offices in Shanghai and North America, Europe and the UK through our Calibre Scientific sister companies. Reagecon employs 100 people, 50% are chemistry or science graduates and most are involved in the development, production, testing, quality control and sales & marketing of over 6,000 product references that we currently produce. We have a very active R&D programme and develop and bring to market many hundreds of new standards, every year.

All Reagecon manufactured products are underpinned by and demonstrate our position as a centre of excellence in the science of Metrology. Product is manufactured, tested, and certified under the applicable ISO/IEC 17025 (A2LA Ref: 6739.03) or ISO/IEC 17034 (A2LA Ref: 6739.01) accreditation or ISO/IEC 17025 (A2LA Ref: 6739.02) for Calibration, in one of our 20 specially equipped laboratories.

The resulting product is classified within one of 54 product families, these families are then grouped and promoted under 7 main product headings, as listed below:-

- Electrochemistry Standards
- Cation and Anion Standards
- Pharmacopoeia Reagents and Standards
- Physicochemical Standards
- Total Organic and Inorganic Carbon Standards
- Volumetric Solutions for Titration
- Customised Standards and Reagents



## Analytical Volumetric Solutions & Indicators

#### Summary of Features & Benefits

- $\checkmark$  High specification:  $\pm 0.1\% / \pm 0.2\%$
- Test method accredited to ISO/IEC 17025
- Stability is proven (either opened or unopened) for the shelf life of this product.
- Consistency of product -Independent, Traceable, Certified
- Widest range of values and pack options available in the market

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- Ready to Use
- Certificates of Analysis and Safety Data Sheets available online

Reagecon produces an extensive range of high-quality Titrants (Analytical Volumetric Solutions). These products are standardised and factorised against a primary standard, where such a primary standard exists, and certified to a specification of  $\pm$  0.1% in the case of common aqueous titrants or  $\pm$  0.2% in the case of less commonly used Titrants or Non-aqueous Titrants.

Reagecon as a producer, that makes such products in large quantities on a regular basis, has extensive product knowledge and expertise and will invariably achieve a specification of a higher order of magnitude, than most laboratories can achieve in-house.

As well as Analytical Volumetric Solutions being of extreme importance in achieving correct results and proving that correctness for both manual and automatic titrations, Indicators are extremely important in manual titrations. In the case of manual titrations, indicator, titrant or analyte change of colour is by far the most important method of end point detection. Therefore, the availability of a wide selection of Indicators is an integral part of any offering of Analytical Volumetric Solutions. Reagecon carries by far the most extensive offering of both Indicators and Titrants available in the marketplace.

#### The Principle of Titrimetry

Titrimetry or measurement by titration includes a set of widely used analytical techniques, some of which have been in widespread use for almost 200 years. Volumetric titration dates back at least to the work of French chemist Gay-Lussac, who devised a method in 1835 to determine the purity of Silver, using standardised Sodium Chloride as the titrant.

The principle of all titrimetry involves the determination of the quantity of the reagent of known concentration (titrant), that is required to react completely with an unknown analyte. Volumetric titrimetry involves measuring the volume of the solution of known concentration (titrant) consumed, gravimetric titrimetry measures the mass of the reagent consumed and coulometric titration measures a direct electrical current of known magnitude that consumes the analyte. In coulometry, the time it takes to complete the electrochemical reaction, is the measurand.

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An analytical volumetric solution (also called titrant, standard titrant or standard solution) is a reagent of known concentration that is added from a burette or other dispensing apparatus to a sample (analyte) until a reaction between the two liquids is judged to be complete. This completeness (end point) is usually observed in a manual titration by the production of a physical change read visually as the titrant is added to the analyte. Such a change may include an appearance, disappearance or change of colour or appearance/disappearance of turbidity (cloudiness).

Nowadays, instruments are widely used to detect the end points by detection of any of several properties or characteristics of the analyte solution including colour, turbidity, temperature, refractive index, potential difference, current or conductivity. In simple terms titrimetry is broadly divided into two main classifications - manual and instrumental - irrespective of how the end point is detected. In the case of manual titrations, indicator, titrant or analyte change of colour is by far the most important method of end point detection. Therefore, the availability of a wide selection of indicators is an integral part of any offering of Analytical Volumetric Solutions. This compendium carries by far the most extensive offering of both indicators and titrants available in the marketplace. The end point in automatic titration is indicated most commonly by a change in potential of an electrode that responds to the concentration of the reagent or the analyte.

#### Analysis by titration brings a large number of benefits to the analyst including the following:

- Relatively easy to perform (although high accuracy manual titration requires practice, dexterity, experience and sound judgement)
- Rapid, cheap and versatile
- Accurate, reproducible, traceable and comparable

Furthermore, titration reactions should exhibit defined stoichiometry, be quantitative, establish equilibrium that is definite and fast, and provide unambiguous results.

#### Types of Titration Reactions

#### Acid/Base Reactions (also called neutralisation titrations)

These are used to determine either the amount of acid/base in an analyte or substances that can be converted to an acid/base. They may also sometimes be used to track the progress of chemical reactions that produce or consume hydrogen ions. The titrants are always strong acids or bases and include hydrochloric acid, perchloric acid, sulphuric acid, sodium hydroxide, potassium hydroxide and sometimes barium hydroxide. Weak acids or bases are not used because they react incompletely with the analyte. The colour indicator used in an acid base titration is a weak acid/ base itself which in its undissociated form differs in colour from its conjugate acid or base form. Typical elements suitable to this type of titration method include carbon, nitrogen, chlorine, bromine and fluorine. Pretreatment of these elements converts the element to an inorganic acid or base that is then titrated. An example is nitrogen which occurs in a wide range of forms both organic, inorganic or as a constituent of biological materials. Therefore, a methodology for nitrogen measurement in amine groups such as the Kjeldahl method is extremely important in determining the protein content in grains, meats, and other human or animal foodstuffs. In addition to amines, others like esters and hydroxyl functional groups can also be determined. In addition, inorganic compounds such as carbonates, ammonium salts and several other NOx species can be determined.

#### **Fields of Application**

- Acid content in wine, milk, ketchup, fruit juice (etc.)
- Content of HCl, HNO3, H2SO4, NaOH, KOH
- Alkalinity determination in water
- TAN and TBN in petroleum products, edible or inedible oils and fats
- Ø Determination of boric acid in cooling fluids of nuclear power stations
- Ø Determination of free or total acidity in plating baths
- Ø Determination of active ingredients in drugs or raw materials for the pharmaceutical industry
- Total nitrogen determination by Kjeldahl
- Wide range of inorganic, organic or biological species that possess inherent acidic or basic properties
- Use of chemical treatment that converts an analyte to an acid or base followed by titration with standardised strong acid or base

#### **Oxidation/Reduction Titrations**

These titrations may be performed manually or potentiometrically. In manual titrations, if indicators are used, they change colour upon being oxidized or reduced, independently of the chemical nature of the titrant or analyte. Instead, they depend on changes in the electro potential of the oxidation reduction system.

#### Examples of such indicators include:

- Iron (III) complexes of orthophenothrolines
- Starch solutions
- Potassium thiocyanate

The principle of this type of titration involves a reaction between an oxidising and reducing pair, e.g. titration of iron (II) with cerium (IV) sulphate

#### Oxidising agents (examples)

- Iodine (Iodometry), potassium dichromate, potassium permanganate, potassium bromate, cerium (IV) ammonium nitrate, cerium (IV) ammonium sulphate, cerium (IV) hydrogen sulphate, cerium hydroxide, chlorine

#### *Reducing agents (examples)*

- Sodium thiosulphate, oxalic acid, iron ammonium (II) sulphate (Mohr's salt), hydrogen peroxide, phenyl arsine oxide (PAO), iron (II) ethylene diamine sulphate

#### **Fields of Application**

- Environment
  - COD of water
  - Oxidation capacity of water by permanganate
- Food and Beverage
  - Determination of free and total SO2 in water, wine, alcohol, dried fruit etc
- Pharmaceuticals
  - Vitamin C determination
  - Surface treatment
  - Titration of copper or tin using iodine
  - Titration of chromium (VI)
- Petrochemicals
  - Determination of water in hydrocarbons

#### **Complexometric Titrations**

Complexometric reactions have many applications in chemical analysis and in science in general. Their use in titrometry is a very important one of these applications. The reaction end point is detected either potentiometrically or manually using an indicator, whereby, a metal ion reacts appropriately with a ligand to form a complex.

EDTA is the most widely used titrant in complexometric reactions although the use of other chemicals similar to EDTA are described in the literature; e.g. nitrilotriacetic acid. Generally, organic dyes that form complexes with metal ions to form chelates are used as indicators, a commonly used one being Eriochrome Black T. Methods have been developed, validated and published for detection or quantification of almost every metal in the periodic table with the exception of the Alkali metals using EDTA complexation. This includes methods for at least 40 metals developed in our metals laboratory in Reagecon, with more at development or validation stage.

This methodology is regularly used to determine the concentration of divalent cations such as calcium, magnesium, copper, lead, zinc, cadmium, aluminum

#### **Fields of Application**

- Environment
  - Determination of chloride in water
- Food and beverage
  - Determination of chloride in many finished products (cooked meats, dairy products, etc.)
- Precious metals
  - Determination of silver
- Pharmaceuticals
  - Titration of halides

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## Total Acid Number / Total Base Number Standards & Reagents

#### Summary of Features & Benefits

#### ASTM D664

- Produced in accordance with ASTM D664.
- Standards are traceable to NIST
- Test method accredited to ISO/IEC 17025
- Interpretation of the second secon measurement which is linked to the TAN value and is expressed as a percentage of that value
- The products have stable proven stability
- Interview of the section of the s potassium hydroxide up to 4.5 mg/grams of potassium hydroxide
- Presented in convenient pack sizes

#### **ASTM D2896**

Product No:

ate:

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- Produced in accordance with ASTM D2896
- Standards are traceable to NIST
- The test method is accredited
- The method has a very low uncertainty of measurement of 1% irrespective of value with proven stability.
- 8 different standard values are available, ranging from 1 mg/gram of Potassium Hydroxide right through to 70 mg/gram of Potassium Hydroxide.
- Presented in convenient pack sizes

Reagecon has a complete offering of standards and reagents for both TAN and TBN, irrespective of the method used. These include titrants, standards, buffers and electrolytes for the major ASTM methods. The suite of products extend to titrants required for back titration and the potassium hydrogen phthalate used to re-standardise or re-factor the titrants.

This offering which is high quality and complete, can afford our loyal customers the best set of products, service and customer care available from any supplier.

These products for Total Acid Number (TAN) and Total Base Number (TBN) are used in procedures to test and control the acidic or basic constituents in petroleum, lubricants, biodiesel or blends of biodiesel.

#### Total Acid Number (TAN)

The procedures for the measurement of this parameter (laid down in various ASTM methods) vary depending on sample solubility in materials such as Toluene or Propan-2-ol, the dissociation constants of the acids in water, or the nature of the test sample. Therefore, the methodology used for lubricants maybe be different from the methodology used for biodiesel. In new and used oils the constituents that maybe considered to have acidic characteristics include organic acids, inorganic acids, esters, phenolic compounds, lactones, resins, salts of heavy metals, acid salts of polybasic acids, and additives such as inhibitors and detergents.

The test method is used to indicate relative changes that occur in oil during use under oxidising conditions regardless of the colour or other properties of the oil. The method is also used as a guide in the quality control of lubricating oil formulations or as a measure of lubricant degradation. It is not intended to measure an absolute acidic property that can be used to predict performance of oil under working conditions. There is no known relationship between corrosion of bearings and acid number. The methodology of performing the test involves dissolving the sample in a titration solvent and titrating potentiometrically as an acid/base titration with alcoholic potassium hydroxide.

#### Total Base Number (TBN)

The constituents of oils and lubricants that may be considered to have basic characteristics include organic bases, inorganic bases, amino compounds, salts of weak acids (soaps), basic salts of poly acidic bases and salts of heavy metals. The test methodology involves dissolving the sample in an anhydrous mixture of chlorobenzene/glacial acetic acid and titrating potentiometrically with a solution of perchloric acid in glacial acetic acid. Both new and used petroleum products can contain basic constituents that are present as additives. The test is sometimes used as a measure of lubricant degradation but any condemning limits based on the test must be established on an individual basis. Reagecon offers a selection of Solvents, Titrants, Standards, Buffers and Electrolytes specifically formulated for the testing of TAN and TBN using ASTM methods D664 and D2896 respectively.





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