

PROVA 1

Dovendo organizzare e gestire lo svolgimento di una attività pratica, prova di laboratorio chimico, denominata **“Determinazione del titolo dell’H₂O₂”** mediante una titolazione con permanganato di potassio, KMnO₄, per 25 studenti all’interno di una struttura dotata di 20 postazioni individuali a banco e di 8 postazioni individuali sotto cappa, commentare almeno 3 degli aspetti sottoelencati al punto 1. Inoltre, svolgere il calcolo stechiometrico riportato al punto 2.

1.

- Discutere il principio del metodo e il campo di applicazione;
- Individuare la strumentazione, i reattivi e gli accessori necessari;
- Spiegare come si procede per la predisposizione della strumentazione, dei reattivi e degli accessori necessari per l’esecuzione della prova;
- Discutere eventuali interferenze e cause di errore;
- Spiegare come si raccolgono le informazioni di sicurezza necessarie per lo svolgimento dell’esperienza e come si procede per etichettare i contenitori dei reattivi iniziali e dei prodotti finali.

2.

Determinazione del titolo di una soluzione di KMnO₄ circa 0.1 N

Stimare la massa di ossalato di potassio, K₂C₂O₄ (MM = 166.22 g/mol), necessaria per titolare 25 mL di una soluzione di KMnO₄ circa 0.1 N (MM = 158.034 g/mol).

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Atomic Absorption Spectroscopy in Analytical Chemistry

Atomic absorption spectroscopy (AAS) is an analytical technique used to measure the concentration of specific elements in a sample. It is widely applied in fields such as environmental analysis, food safety, pharmaceuticals, and metallurgy due to its accuracy and sensitivity.

The basic principle of AAS involves the absorption of light by free atoms in the gas phase. A sample is first vaporized in a flame or graphite furnace, breaking it down into individual atoms. A light source, typically a hollow cathode lamp, emits radiation at wavelengths specific to the element being analyzed. As the light passes through the vaporized sample, the atoms of the target element absorb the light at their characteristic wavelength.

The amount of absorbed light is proportional to the concentration of the element in the sample. A detector measures the reduction in light intensity, and the results are displayed as an absorption value. Using calibration curves, the concentration of the element can be quantified accurately.

AAS is highly sensitive, capable of detecting trace amounts of elements, often in parts per million (ppm) or even lower. It is particularly useful for analyzing metals such as lead, cadmium, and mercury. Its precision and reliability make atomic absorption a cornerstone of modern analytical chemistry.



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PROVA 2

Dovendo organizzare e gestire lo svolgimento di una attività pratica, prova di laboratorio chimico, denominata **“Determinazione della durezza totale”** di un campione di acqua mediante titolazione complessometrica per 18 studenti in un laboratorio dotato di 20 postazioni individuali a banco e di 8 postazioni individuali sotto cappa, commentare almeno 3 degli aspetti riportati al punto 1 e svolgere il calcolo stechiometrico riportato al punto 2.

La determinazione della durezza totale dell’acqua si basa sulla formazione dei complessi tra i cationi metallici Mg^{2+} e Ca^{2+} con la forma anionica dell’acido etilendiamminotetraacetico (EDTA) in ambiente basico tamponato.

1.

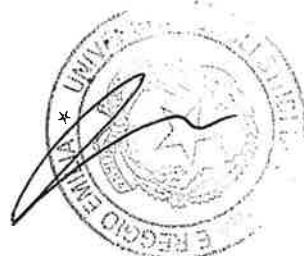
- Discutere il principio del metodo e il campo di applicazione
- Spiegare come si procede per la predisposizione delle apparecchiature e la preparazione dei reattivi per l’esecuzione della prova.
- Spiegare come si procede per la verifica della concentrazione della soluzione titolante di EDTA in assenza di soluzioni commerciali a titolo noto.
- Discutere eventuali interferenze e cause di errore.
- Spiegare come si raccolgono le informazioni di sicurezza necessarie per lo svolgimento dell’esperienza e come si procede per etichettare i contenitori dei reattivi iniziali e dei prodotti finali.

2.

Calcolo stechiometrico

Esprimere la durezza in gradi francesi di un campione di acqua sapendo che per titolare 100 mL di campione sono necessari 20 mL di una soluzione acquosa di EDTA 0.01 M. Un grado francese corrisponde a 0.01 g/L di $CaCO_3$ ($MM = 100.09$ g/mol). Massa atomica Ca: 40.08 g/mol.

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Ultraviolet-visible spectroscopy

Ultraviolet-visible spectrophotometry (UV-Vis or UV-VIS) refers to absorption spectroscopy or reflectance spectroscopy in part of the ultraviolet and the full, adjacent visible regions of the electromagnetic spectrum. Being relatively inexpensive and easily implemented, this methodology is widely used in diverse applied and fundamental applications. The only requirement is that the sample absorb in the UV-Vis region, i.e. be a chromophore. Absorption spectroscopy is complementary to fluorescence spectroscopy. Parameters of interest, besides the wavelength of measurement, are absorbance (A) or transmittance (%T) or reflectance (%R), and its change with time.

A UV-Vis spectrophotometer is an analytical instrument that measures the amount of ultraviolet (UV) and visible light that is absorbed by a sample. It is a widely used technique in chemistry, biochemistry, and other fields, to identify and quantify compounds in a variety of samples.

UV-Vis spectrophotometers work by passing a beam of light through the sample and measuring the amount of light that is absorbed at each wavelength. The amount of light absorbed is proportional to the concentration of the absorbing compound in the sample.

Most molecules and ions absorb energy in the ultraviolet or visible range, i.e., they are chromophores. The absorbed photon excites an electron in the chromophore to higher energy molecular orbitals, giving rise to an excited state. For organic chromophores, four possible types of transitions are assumed: $\pi-\pi^*$, $n-\pi^*$, $\sigma-\sigma^*$, and $n-\sigma^*$. Transition metal complexes are often colored (i.e., absorb visible light) owing to the presence of multiple electronic states associated with incompletely filled d orbitals.



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PROVA 3

Dovendo organizzare e gestire lo svolgimento di una attività pratica, prova di laboratorio chimico, denominata **“Determinazione del nichel”** per via gravimetrica per 40 studenti in un laboratorio dotato di 20 postazioni individuali a banco e di 14 postazioni individuali sotto cappa, commentare almeno 3 degli aspetti sottoelencati al punto 1. Inoltre, svolgere il calcolo stechiometrico riportato al punto 2.

1.

- Discutere il principio del metodo e il campo di applicazione
- Individuare la strumentazione, i reattivi e gli accessori necessari.
- Spiegare come si procede nella predisposizione della strumentazione, dei reattivi e degli accessori necessari.
- Discutere eventuali interferenze e cause di errore
- Spiegare come si raccolgono le informazioni di sicurezza necessarie per lo svolgimento dell’esperienza e come si procede per etichettare i contenitori dei reattivi iniziali e dei prodotti finali.

2.

Calcolo stechiometrico

Calcolare il volume di NH_3 ($\text{MM} = 17.03 \text{ g/mol}$) al 32 % M/M (densità = 0.886 g/mL) necessario per preparare 500 mL di una soluzione acquosa di NH_3 avente concentrazione pari a 3 mol/L .

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Gas Chromatography in Analytical Chemistry 209 words

Gas chromatography is a powerful analytical technique used to separate, identify, and quantify volatile compounds in complex mixtures. The method relies on the distribution of sample components between a mobile gas phase and a stationary phase within a column. When the sample is injected into the heated column, its constituents vaporize and travel through the column at different rates depending on their interactions with the stationary phase. A detector at the column's end records the elution of each compound, producing a chromatogram that displays distinct retention times characteristic of each analyte.

In an educational laboratory, gas chromatography serves as an excellent tool for demonstrating the principles of separation and analysis, effectively linking theoretical knowledge with practical applications. Calibration with known standards allows for precise quantification of unknown sample concentrations, reinforcing important concepts such as retention time and peak integration.

The technical collaborator plays a crucial role in the educational setting by preparing samples, maintaining the instrument, and calibrating the system to ensure accurate and reproducible results. Additionally, the collaborator is responsible for managing safety protocols and ensuring that all operational procedures are followed. This hands-on involvement not only guarantees the efficient functioning of the laboratory but also enhances students' learning experiences by providing practical insights into modern analytical techniques.



A handwritten signature in blue ink is located to the right of the university seal. The signature consists of two stylized, flowing lines that intersect and curve, forming a unique, personal mark.

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PROVA 4

Dovendo organizzare e gestire lo svolgimento dell'esperienza di laboratorio denominata: "Determinazione spettrofotometria UV-vis del ferro" in un campione di acqua per 30 studenti in un laboratorio dotato di 20 postazioni individuali a banco, 14 postazioni individuali sotto cappa, 3 spettrofotometri UV-vis mono raggio ed uno spettrofotometro a doppio raggio, commentare almeno 3 degli aspetti riportati al punto 1. Inoltre, svolgere il calcolo stechiometrico riportato al punto 2.

Il metodo per la determinazione del Fe in acqua si basa sulla formazione del complesso tra il Fe (II) e la o-fenantrolina in ambiente acido. La costante di formazione del complesso a 25°C è: $K = 2.5 \cdot 10^6$.

1.

- Discutere il principio del metodo e il campo di applicazione
- Spiegare come si procede nella predisposizione della strumentazione, dei reattivi e degli accessori necessari.
- Spiegare come si effettua la scelta della lunghezza d'onda di analisi e la preparazione della curva di calibrazione
- Discutere eventuali interferenze e cause di errore
- Spiegare come si raccolgono le informazioni di sicurezza necessarie per lo svolgimento dell'esperienza e come si procede per etichettare i contenitori dei reattivi iniziali e dei prodotti finali.

2.

Calcolo stechiometrico

Determinare la massa di solfato ferroso esaidsrato, $\text{FeSO}_4 \cdot 6\text{H}_2\text{O}$ ($\text{MM} = 392.14 \text{ g/mol}$), per preparare la soluzione madre necessaria per costruire la curva di taratura per la determinazione del Fe nell'intervallo di concentrazione: $1 < \text{Fe (mg kg}^{-1}) < 5$. Massa atomica Fe = 55.9 g/mol.

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High-Performance Liquid Chromatography (HPLC)

High-Performance Liquid Chromatography (HPLC) is an analytical technique widely used in chemistry and biochemistry to separate, identify, and quantify components in a mixture. It relies on the principles of liquid chromatography, where a liquid mobile phase carries the sample through a column packed with a solid stationary phase. The different components of the sample interact differently with the stationary phase, causing them to elute at different times, known as retention times.

HPLC systems consist of a pump, an injector, a column, a detector, and a data processing unit. The pump moves the mobile phase through the system at a constant flow rate, while the injector introduces the sample into the stream. The column is the heart of the system, where the actual separation occurs. Detectors, such as UV-Vis, fluorescence, or mass spectrometers, are used to identify and quantify the compounds based on their specific responses.

HPLC is highly precise, reproducible, and capable of analyzing complex mixtures with high resolution. It is commonly used in pharmaceutical industries for drug purity testing, in environmental monitoring, and in food quality control. Depending on the nature of the analysis, different modes of HPLC can be employed, such as reverse-phase, normal-phase, ion-exchange, or size-exclusion chromatography.

Overall, HPLC is a versatile and essential tool in modern analytical laboratories.

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PROVA 5

Dovendo organizzare e gestire lo svolgimento di una attività pratica, prova di laboratorio chimico, denominata **“Determinazione mediante titolazione potenziometrica della concentrazione di una base debole monoprotica”** per 40 studenti in un laboratorio dotato di 20 postazioni individuali a banco, di 14 postazioni individuali sotto cappa e di 15 pH-metri. Commentare almeno 3 degli aspetti riportati al punto 1. Inoltre, svolgere il calcolo stechiometrico riportato al punto 2.

1.

- Discutere il principio del metodo e il campo di applicazione;
- Spiegare come si procede nella predisposizione delle apparecchiature e nella preparazione dei reattivi;
- Discutere le operazioni di calibrazione della strumentazione e standardizzazione dei reagenti necessari;
- Discutere eventuali interferenze e cause di errore;
- Spiegare come si raccolgono le informazioni di sicurezza necessarie per lo svolgimento dell'esperienza e come si procede per etichettare i contenitori dei reattivi iniziali e dei prodotti finali.

2.

Calcolo stechiometrico

Calcolare il volume di una soluzione acquosa di HCl (MM = 36.461 g/mol) al 35.0 % M/M (densità = 1.175 g/mL) necessario per preparare 500 mL di una soluzione acquosa di HCl avente concentrazione pari a 0.25 mol/L.

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Fourier Transform Infrared Spectroscopy (FT-IR)

Fourier Transform Infrared Spectroscopy (FT-IR) is a powerful analytical technique used to obtain the infrared spectrum of absorption or emission of a solid, liquid, or gas. It provides information about the molecular structure and chemical composition of a sample by measuring how it absorbs infrared (IR) radiation at different wavelengths.

In FT-IR, a beam of infrared light is passed through a sample. Some of the light is absorbed, and the rest is transmitted. The resulting signal, called an interferogram, is collected over a range of wavelengths simultaneously. A mathematical process known as the Fourier Transform is then applied to convert the interferogram into an actual spectrum, showing the intensity of absorption as a function of wavelength or frequency.

Each molecule has a unique infrared spectrum, acting like a "molecular fingerprint." This allows FT-IR to be used for both qualitative and quantitative analysis. It is commonly used in chemistry, pharmaceuticals, forensics, materials science, and environmental studies.

FT-IR spectroscopy is valued for its speed, accuracy, and minimal sample preparation. It can identify functional groups, detect impurities, and monitor chemical reactions. Advanced techniques like ATR (Attenuated Total Reflectance) allow direct analysis of samples with little or no preparation.

Overall, FT-IR is an essential tool for non-destructive molecular analysis.

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