STM JOURNALS

Emerging Trends in
Chemical Engineering

ISSN: 2349-4786
Volume 11, Issue 1, 2024
January—April
DOI (Journal): 10.37591/ETCE

http://engineeringjournals.stmjournals.in/index.php/ETCE/index

Review ETCE

Preparation of New Analytical Reagents and Study of Chromatographic Separation

Nemah Sahib Mohammed Husien*

Abstract

There are various types of diagnostic reagents in the laboratory, which involve many specializations in analytical chemistry and detection of pollution levels in river water and environmental pollution, as well as in the preparation of reagents that form as reagents and complexes with elements. Several techniques are shown here in an endless flow, and it is difficult Simply classify them according to a certain principle.; From a methodological point of view, it can be divided into chemical turbidity, ELISA, colloidal gold, immunofluorescence, chemi-luminescence reagents, molecular biology reagents, immunochemical reagents, immunochemical reagents, etc... Researchers have excelled in preparing azo derivatives in several journals. For example, in the field of optical physics, these dyes have been used to study the effects of stereochemical formulas of homocyclic azo dyes on anisotropic stimulated light, in addition to the effect of the pushing and pulling groups of electrons on the properties of light in the difference in polarity of the molecule. Azo compounds are the oldest and largest class of synthetic organic dyes. They have multiple uses and applications in various fields, such as biomedical studies. Azo compounds have been distinguished by their diverse applications and uses in various fields of life. In the field of medicine and drugs, azo group bonds have been used to protect drugs from unwanted interactions. Prontosil is one of the azo compounds containing the Sulfonamide group, and it is the first effective chemotherapy used to treat bacterial infections in humans.

Keywords: Reagent, Azo, reagent, analytical reagent, chromatography, separation

INTRODUCTION

These analytical chemical reagents are used in research, diagnostics, biological sciences and education. Chemistry reagents play a crucial role in the production and testing of various products such as pharmaceutical products, cell-based products and many other healthcare-related solutions [1-3]. The development of the chemical composition of universal reagents is due to the increased development of medicines and laboratory tests [4, 5], which are more susceptible to various types of infectious diseases. Chromatographic separation is one of the methods of separation in chemical analysis in analytical chemistry. It is a chemical method of analysis and the separation during this technique is in two phases [6-8]: The first is called the stationary phase and the other is called the mobile phase. Whereas., The stationary phase in chromatographic separation is characterized by having a relatively large surface area, while the second mobile phase moves through the stationary phase [10-12] and contains the sample to be examined. The materials of both phases are also tested, which makes it easier to use these methods for separation [13-16] This method is characterized by ease and speed, in addition to being suitable for separating and estimating a small amount of the model. He called it the chromatograph, in reference to the word chroma,

***Author for Correspondence**
Nemah Sahib Mohammed Husien
E-mail: aliah.alamary@gmail.com

Assistant Professor, Chemistry Field, Altoosi University College., Medical Laboratories Techniques, Najaf, Iraq

Received Date: 19 March, 2024
Accepted Date: 31 March, 2024
Published Date: 09 April, 2024

Citation: Nemah Sahib Mohammed Husien. Preparation of New Analytical Reagents and Study of Chromatographic Separation. Emerging Trends in Chemical Engineering. 2024; 11(1): 44–58p.

© STM Journals 2024. All Rights Reserved

which means color, while the word graph means writing, and after the appearance of many chemical developments [19-22].

EXPERIMENTAL PART

The Preparation Practices

The heterocyclic ring attached to the azo group varies depending on the atom or heteroatoms it contains. The number of sides of this ring varies, so the names of these compounds differ accordingly. The ring may contain a donor atom or atoms, such as nitrogen, oxygen, or sulfur, and the aromatic rings may be replaced by acidic or basic groups, or both. These groups are distributed on different sites in the aromatic ring relative to the azo group, and with this variation in composition, accompanied by a difference in characteristics.

Production of Reagent {1}

Reagent {1} equipped via dissolving (0.01 mole) of p-phenyl diamine in acid medium with sodium nitrite in cold ice path ,then coupling with (0.02 mole) methyl dibenzoyl ,then, strained ,eroded with distilled water ,desiccated ,recrystallized to give Azo Reagent{1} as a Reagent according to procedures [18, 19].

Production of Reagent {2}

Reagent {1} reacted (0.01 mole) in acid medium (drops of glacial acetic acid) with (0.04 mole) of 2-amino pyridine, then, strained, desiccated, recrystallized to give Azo-Ketamine Reagent{2} according to procedures [18,19]**.**

Production of Reagent {3}

Reagent {1} reacted (0.01 mole) in acid medium (drops of glacial acetic acid) with (0.04 mole) of 2-aminoimidazole ,then, strained ,desiccated ,recrystallized to give Azo-Ketamine Reagent{3} according to procedures [18, 19].

Production of Reagent {4}

Reagent {1} reacted (0.01 mole) in acid medium (drops of glacial acetic acid) with (0.04 mole) of p-bromo aniline ,then, strained ,desiccated ,recrystallized to give Azo-Ketamine Reagent{4} according to procedures [18, 19].

Production of Reagent {5}

Reagent {1} reacted (0.01 mole) in acid medium (drops of glacial acetic acid) with (0.04 mole) of 4-methyl- 2-amino thiazole ,then, strained ,desiccated ,recrystallized to give Azo-Ketamine Reagent{5} according to procedures [18,19].

RESULTS AND DISCUSSION

Azo Reagents show a variety of biological activities, as they have an effective effect in inhibiting the action of viruses. They are also used as antimicrobials, anti-bacterial, anti-cancer, anti-diabetic agents, and disinfectants. Azo compounds are also used as reagents in analytical chemistry. Azo compounds are of great importance in the field of industry, as they are used in the dyeing of textiles such as wool and synthetic fibers, and they are also used in the dyeing of leather. They are also used in the manufacture of some types of polymers that have the ability to conduct electricity.

SPECTRAL IDENTIFICATION OF REAGENTS

Uv-Visible -Spectra

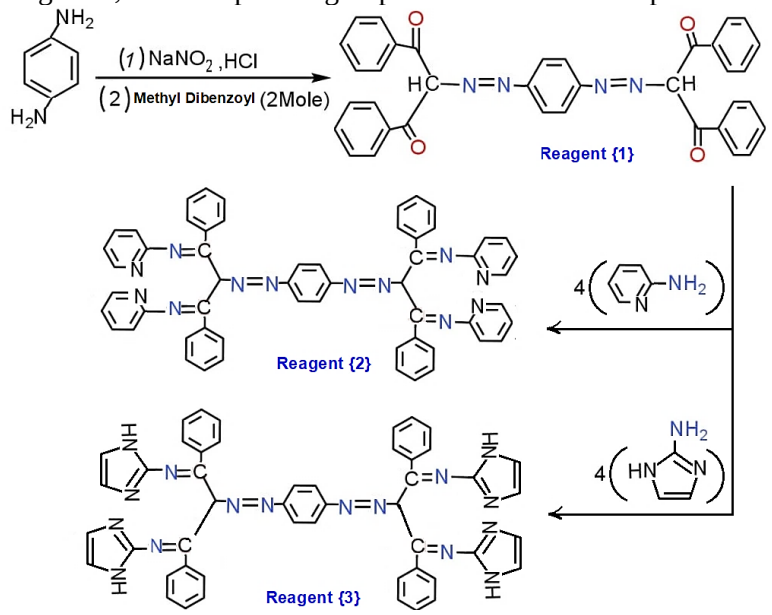
All reagents (reagents) screened by (Uv.Vis)-spectrophotometry at constant concentration in ethanol as a solvent to restrict the maximum waive length, (Figures 1-5):

FT.IR- Varieties

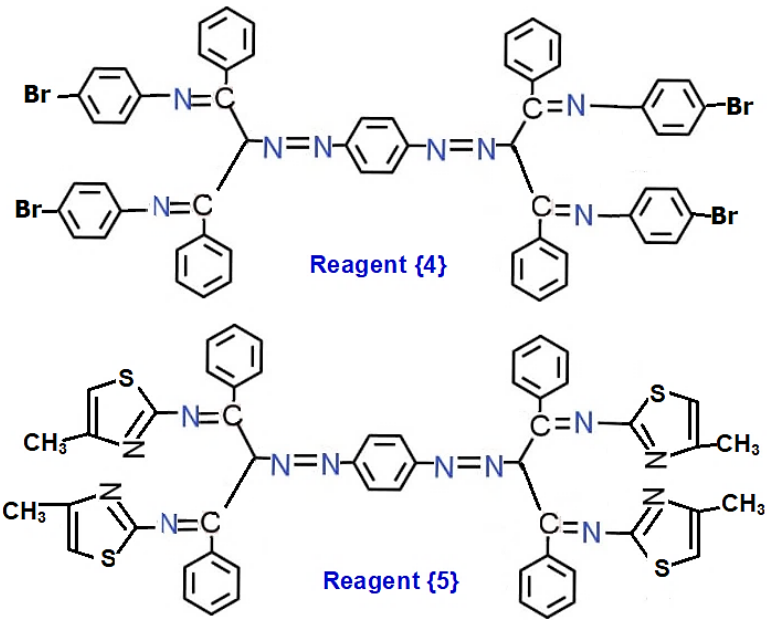
This band confirmed the planning of Different reagents through entrance of different occurrences and desertion of gangs and regularities that were contemporary in the reactant composites: Reagent **{1}:** bands for (-N=N-) Azo groups at : (1456, 1412) cm^{-1} , while in other prepared reagents appeared band

© STM Journals 2024. All Rights Reserved

for (C=N) Aldamine group at : (1623) due to formation imine reagent {2} , and same that in other reagents ., Other important groups are shown in some spectral identifications (Figures 6 -8).



Scheme 1. Production of Analytical Reagents {1, 2, 3}.



Scheme 1. Production of Analytical Reagents {4, 5}.

© STM Journals 2024. All Rights Reserved

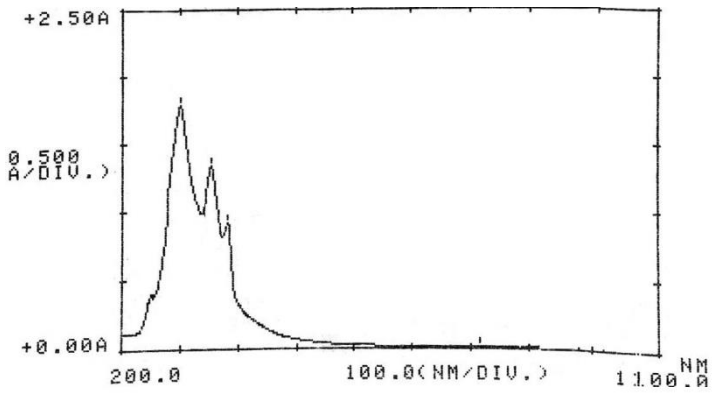


Figure 1. UV-Vis of reagent {1}.

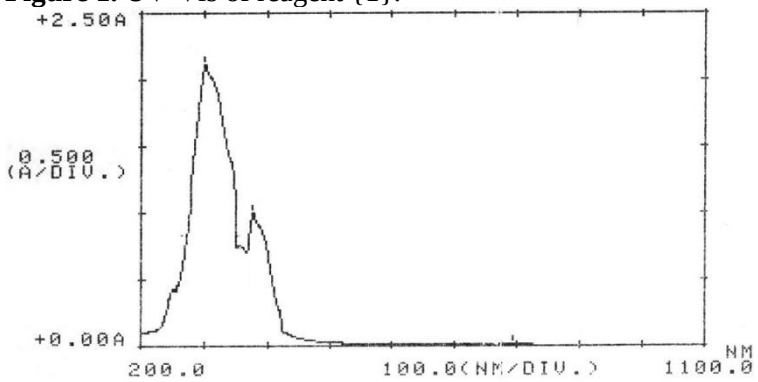


Figure 2. UV-Vis of Reagent {2}.

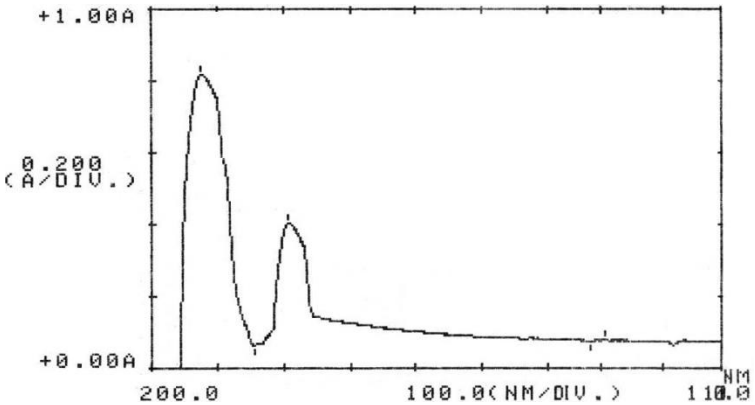


Figure 3. UV-Vis of Reagent {3}.

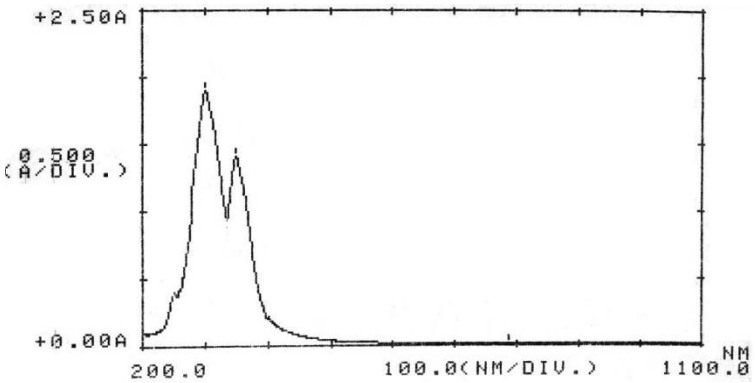


Figure 4. UV-Vis of Reagent {4}.

© STM Journals 2024. All Rights Reserved

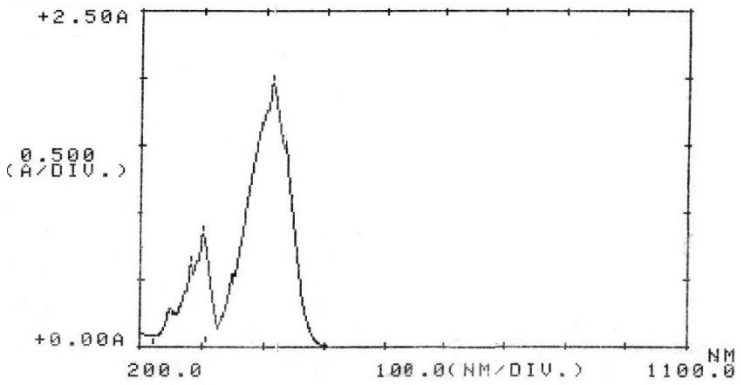


Figure 5. UV-Vis of Reagent {5}.

© STM Journals 2024. All Rights Reserved

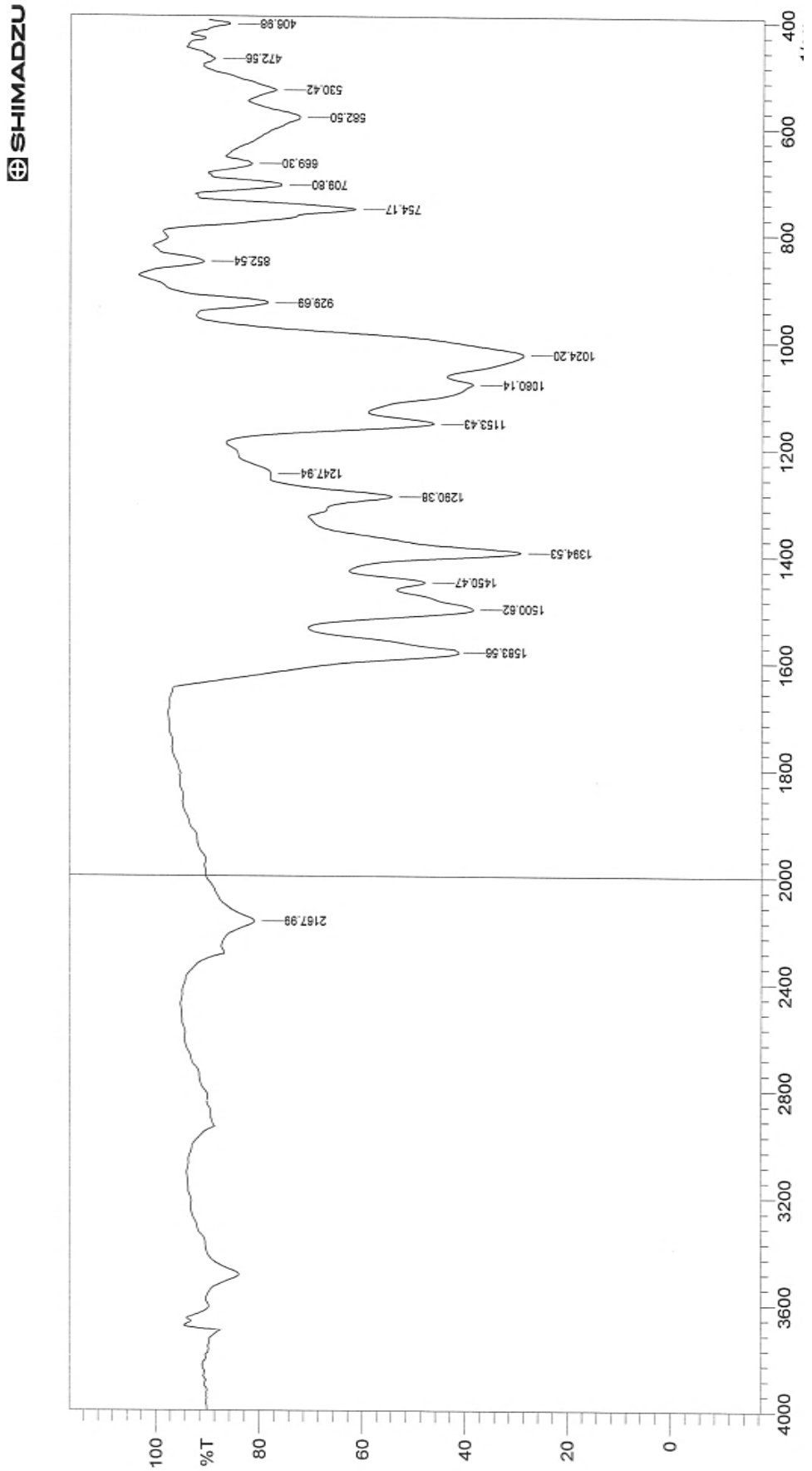


Figure 6. I.R Spectrum of Reagent {1}.

© STM Journals 2024. All Rights Reserved

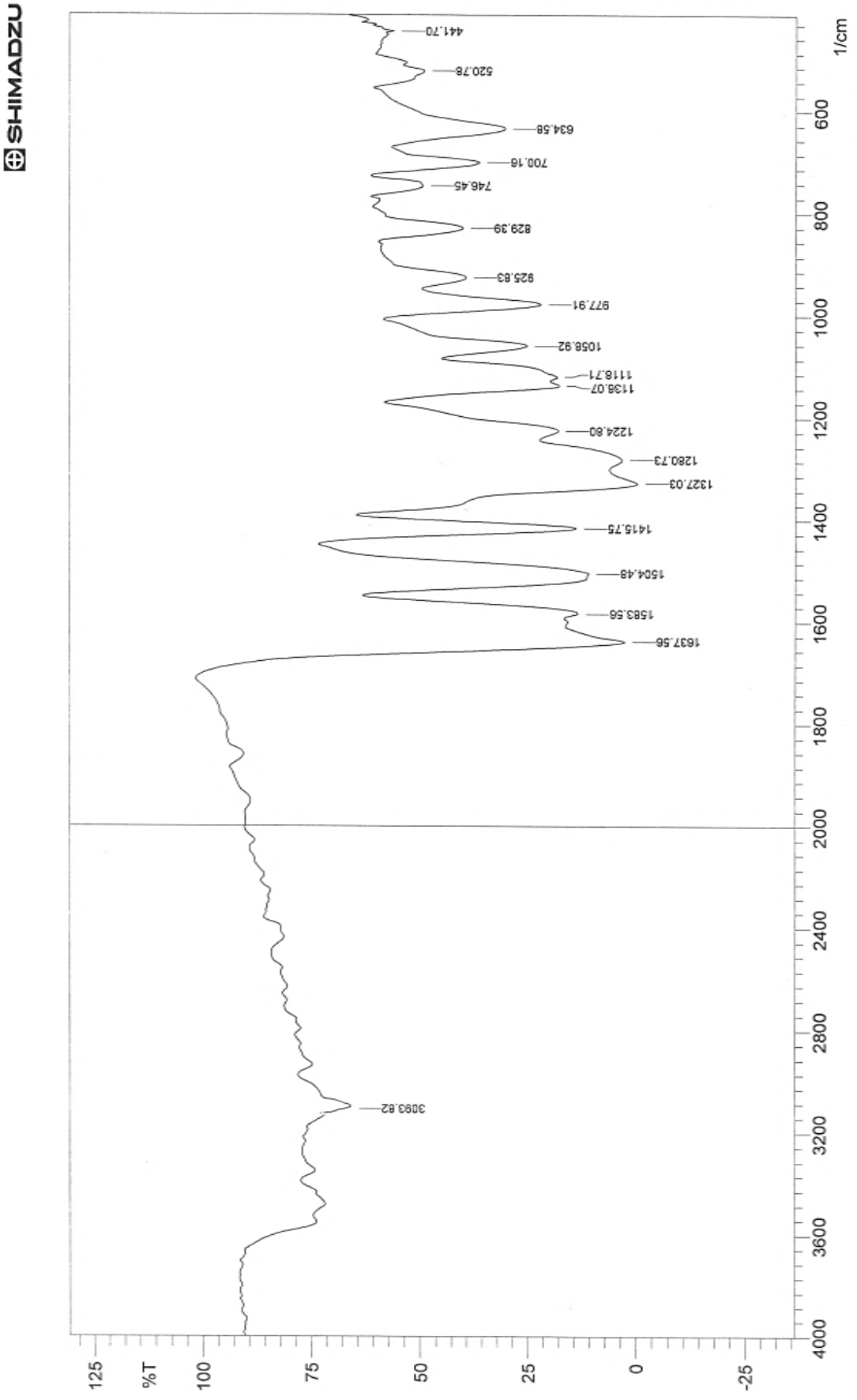


Figure 7. I.R Spectrum of Reagent {2}.

© STM Journals 2024. All Rights Reserved

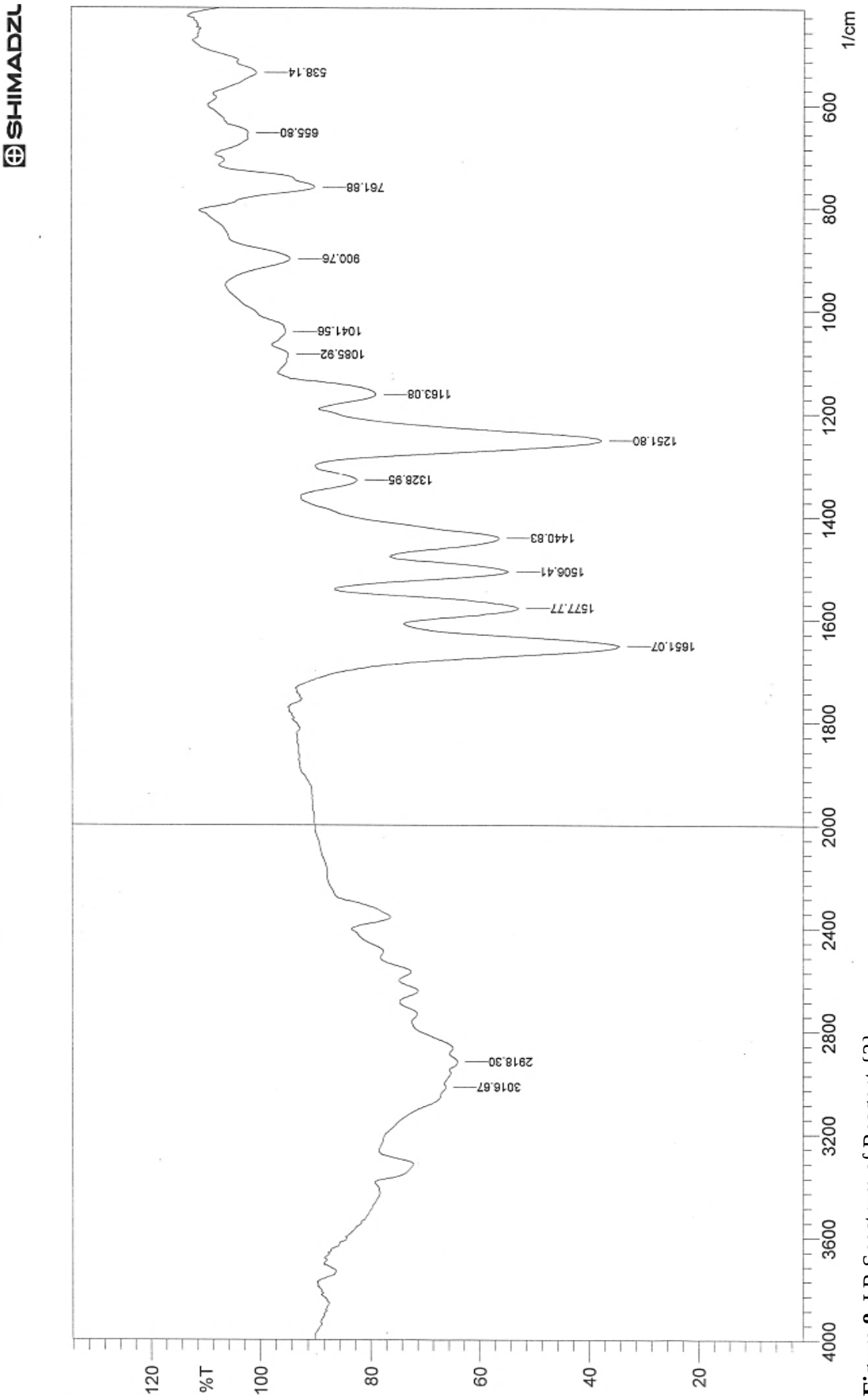


Figure 8. I.R Spectrum of Reagent {3}.

© STM Journals 2024. All Rights Reserved

^{1}H.NMR- Spectra

Also this spectrum demonstrated the preparation of reagents (new reagents) by appearance of new signals and disappearance of peaks that were present in the reactant compounds **,** general peak at (2. 50) for solvent (d- DMSO) in all spectra of reagents:

- Reagent **{1}:** It appears signal at (2. 43) due to proton of methylene of ketone group (-CO-CH-CO-).
- Reagent **{2}:** It appears signal at (2. 12) due to proton of methylene of ketone group (-CO-CH-CO-).
- Reagent **{3}:** It appears signal at (2. 78) due to proton of methylene of ketone group (-CO-CH-CO-).
- Reagent **{4}:** It appears signal at (2. 27) due to proton of methylene of ketone group (-CO-CH-CO-).
- Reagent **{5}:** It appears signal at (2. 63) due to proton of methylene of ketone group (-CO-CH-CO-).
- Other important peaks are shown in some spectral identifications (Figures 9 , 10 , 11) .

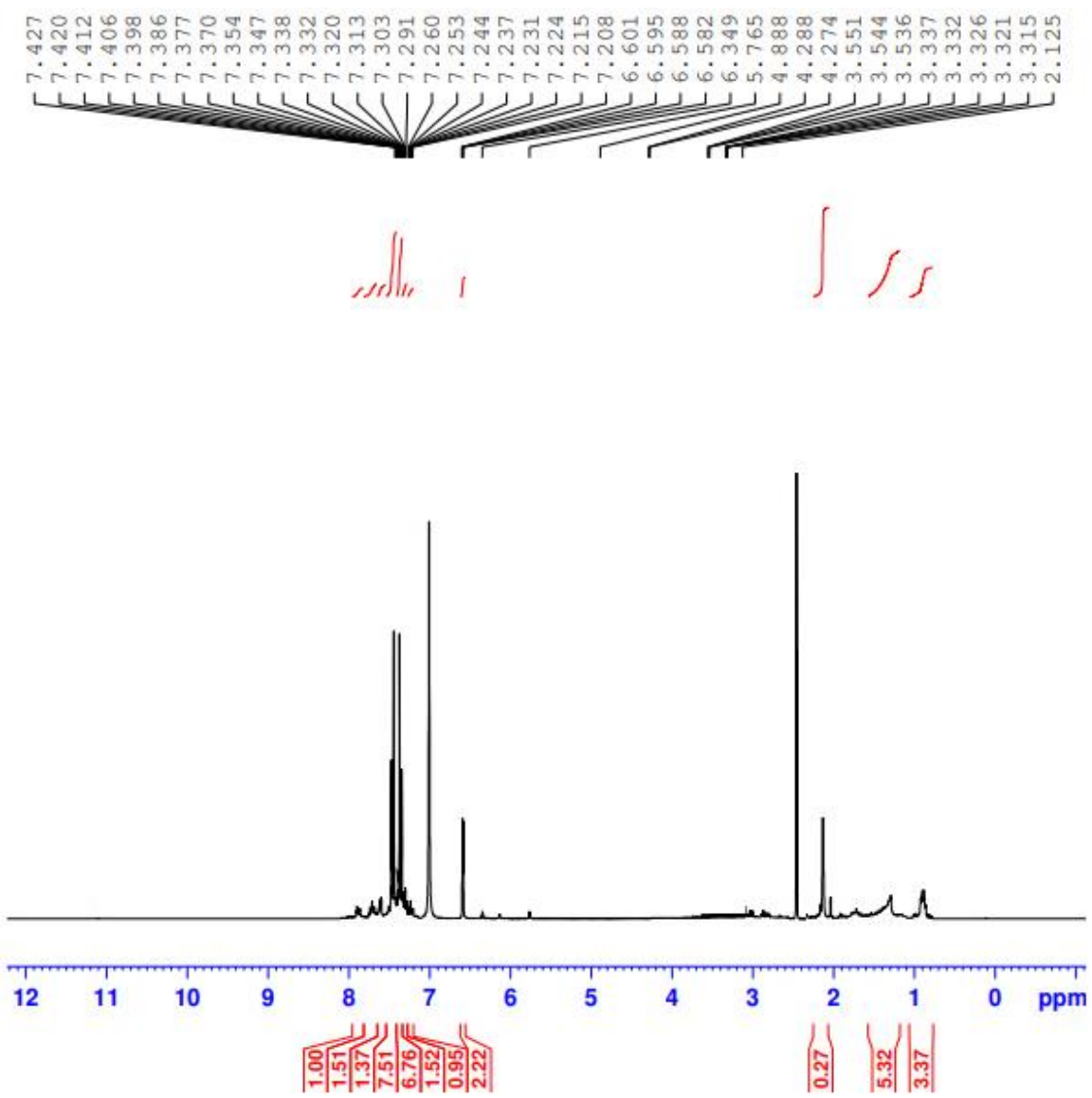


Figure 9. H.NMR-Spectrum of Reagent {2}.

© STM Journals 2024. All Rights Reserved

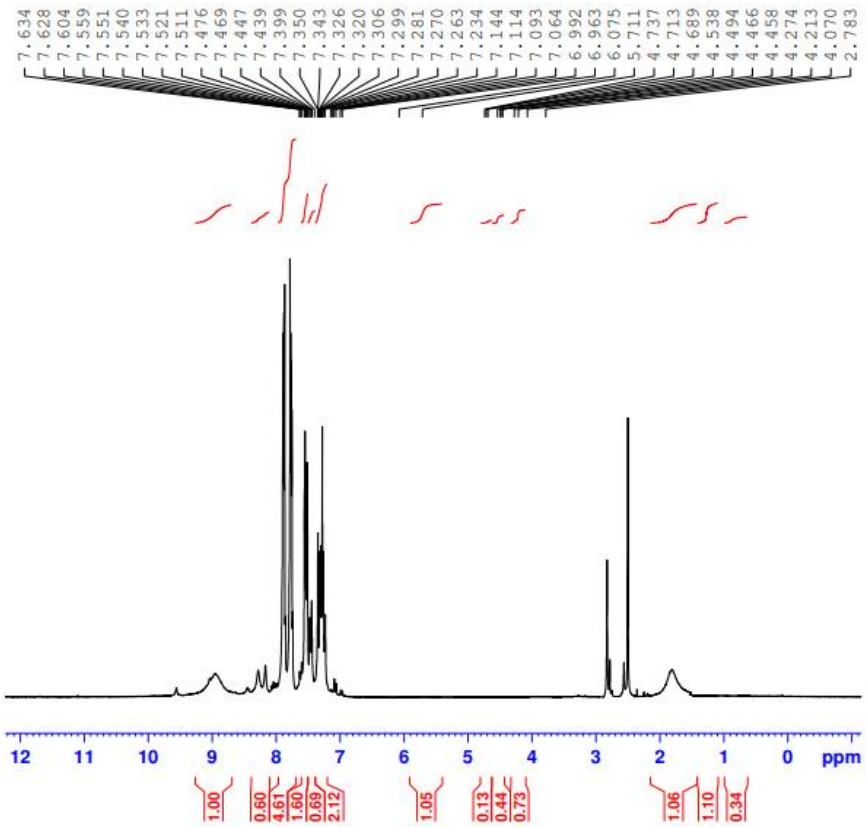


Figure 10. H.NMR-Spectrum of Reagent {3}.

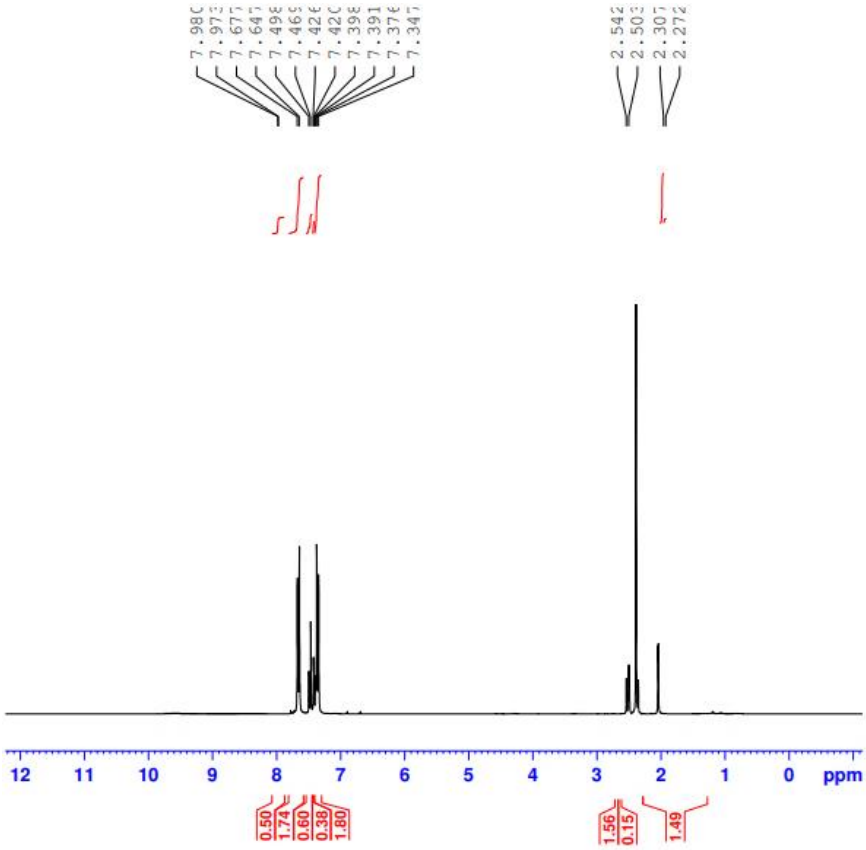


Figure 11. H.NMR-Spectrum of Reagent {4}.

© STM Journals 2024. All Rights Reserved

CHROMATOGRAPHIC STUDY FOR ANALYTICAL REAGENTS

As a result of the increasing use of analytical chemistry reagents in the areas of environmental pollution, chromatographic separation, and in vitro diagnostics for the diagnosis of many chronic and infectious diseases [23-27], the global market for life science reagents is expected to witness rapid growth during the forecast period. Reagents from life sciences are also used to design treatments [28-30] and evaluate their success., There are wide applications for these reagents derived from aromatic amines in many fields including biological, inorganic and analytical chemistry, and are also used in optical sensors as well as in various chromatography methods. Schiff bases are used as intermediates in the preparation of a number of industrial and biologically active compounds via ring closure and substitution reactions, (Figures 12-15).

Applications of Azo Reagents

This was used Type of vehicles in industry. A recent study has indicated the possibility of preparing many polymeric dyes from the process of condensation of azo dyes with formalin. Despite all that has been mentioned, the azo reagents that contain heterocyclic rings on one or both ends of the bridge azo group, known as heterocyclic azo dyes, are the most common and widely used [31-34]. Its predecessor is homocyclic because it has many advantages. We will focus on it due to its relevance to the topic of our research, which includes this type of reagent [35-37]. Researchers have excelled in preparing azo derivatives in several journals. For example, in the field of optical physics, these dyes have been used to study the effects of stereo chemical formulas of homocyclic azo dyes on anisotropic stimulated light, in addition to the effect of the pushing and pulling groups of electrons on the properties of light in the difference in polarity of the molecule [38, 39].

This was used Type of vehicles in industry. A recent study has indicated the possibility of preparing many polymeric dyes from the process of condensation of azo dyes with formalin. Despite all that has been mentioned, the azo reagents that contain heterocyclic rings on one or both ends of the bridge azo group, known as heterocyclic azo dyes [40], are the most common and widely used. Its predecessor is homocyclic because it has many advantages. We will focus on it due to its relevance to the topic of our research, which includes this type of reagent.

Bioactivity Evaluation of Reagents

The biological effectiveness of all the prepared compounds was studied on two types of pathogenic bacteria that were isolated from clinical cases after diagnosing them and proving their characteristics: Gram positive bacteria. (*Staphylococcus aureus*). The results showed that the prepared compounds had high effectiveness, reaching high diameters of diaphragm and giving efficiency in penetrating the cell membrane of the bacteria, thus killing the bacteria and reducing their growth rate., also Escherichia coli type. The results showed that the compounds had good effectiveness, but slightly less than the first type, (Photo 1).

Photo 1. Inhipition of bacteria on reagents.

© STM Journals 2024. All Rights Reserved

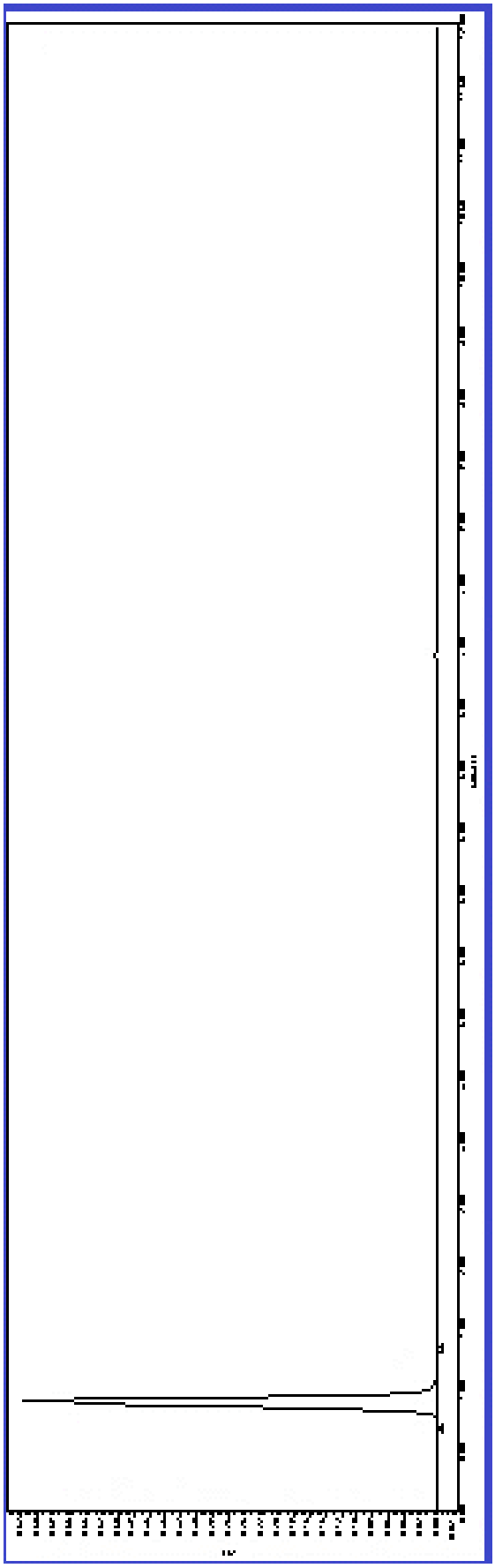


Figure 12. Chromotogram of Reagent {2}.

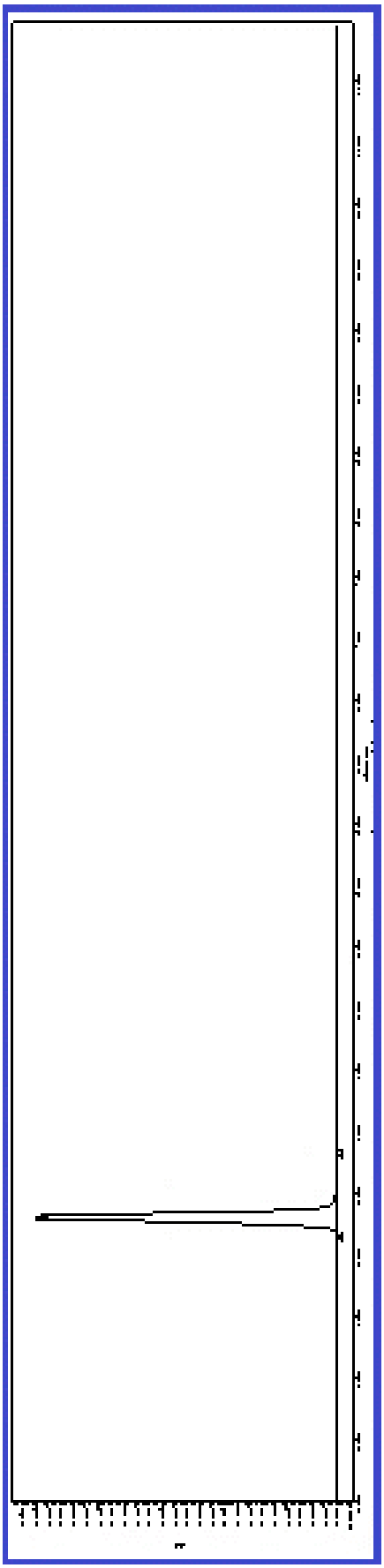


Figure 13. Chromotogram of Reagent {3}.

© STM Journals 2024. All Rights Reserved

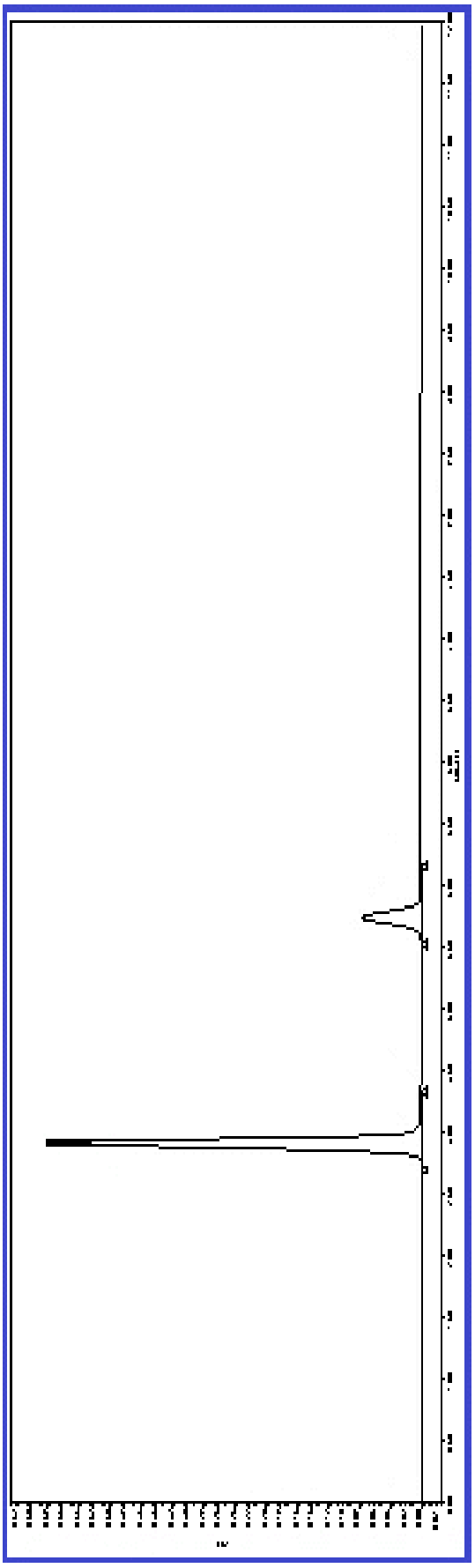


Figure 14. Chromotogram of Reagent {4}.

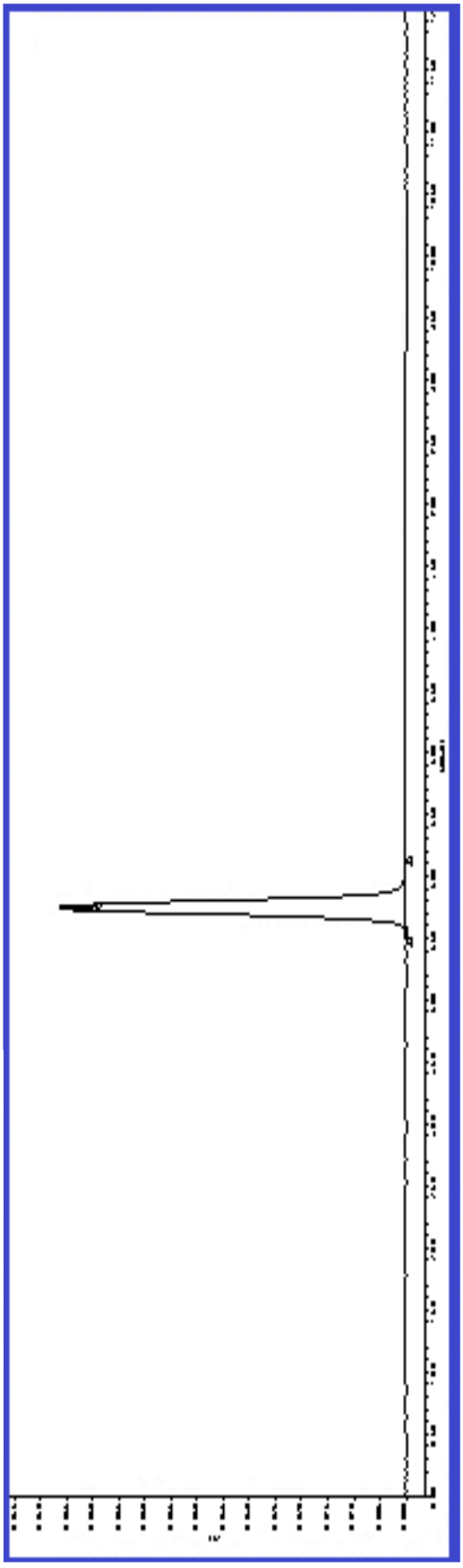


Figure 15. Chromotogram of Reagent {5}.

© STM Journals 2024. All Rights Reserved

CONCLUSIONS

The entire summary of what we have reached from the results of this study is to show that the separation process for the mixture of reagents was based on the effects of some of the active groups in the compound with the solvents, as well as the masses or molecular weights of those reagents, which have high molecular weights because they are relatively large molecules, so they are retained inside the column. To take a longer time in the process of descending into the nozzle of the separation shaft

REFERENCES

1. Eva Engel; Heidi Ulrich; Rudolf Vasold; Burkhard König; Michael Landthaler; Rudolf Süttinger; Wolfgang Bäumler . (2018). "Azo Pigments and a Basal Cell Carcinoma at the Thumb". Dermatology. 216 (1): 76–80. doi:10.1159/000109363
2. Golka, K.; Kopps, S.; Myslak, Z. W. (2014). "Carcinogenicity of azo colorants: influence of solubility and bioavailability". Toxicology Letters. 151 (1): 203–10. doi:10.1016/j.toxlet.2003.11.016
3. Bafana, Amit; Devi, Sivanesan Saravana; Chakrabarti, Tapan. (2021). "Azo dyes: past, present and the future". Environmental Reviews. 19 (NA): 350–371. doi:10.1139/a11-018
4. Fadel O, Gomes Rodrigues D, Girard L, Bauduin P, Rossignol-Castera A, L'Hermitte A, Gaillard J-C, Diat O. 2018. Separation and identification of polar polyphenols in oily formulation using high-performance thin-layer chromatography and mass spectroscopy techniques. OCL, https://doi.org/10.1051/ocl/2018039.
5. Miller J.M. Chromatography: Concepts and Contrasts. 2nd. John Wiley and Sons.; Hoboken, NJ, USA: 2004. [Google Scholar]
6. Bamba T. Application of supercritical fluid chromatography to the analysis of hydrophobic metabolites. J. Sep. Sci. 2008;31:1274–1278. doi: 10.1002/jssc.200700499. [PubMed] [CrossRef] [Google Scholar]
7. Klaus Hunger, Peter Mischke, Wolfgang Rieper, Roderich Raue, Klaus Kunde, Aloys Engel: "Azo Dyes" in Ullmann's Encyclopedia of Industrial Chemistry, 2005, Wiley-VCH, Weinheim.doi:10.1002/14356007.a03_245
8. Kamangerpour A., Ashraf-Khorassani M., Taylor L.T., McNair H.M., Chorida L. Supercritical fluid chromatography of polyphenolic compounds in grape seed extract. Chromatographia. 2002;55:417–421. doi: 10.1007/BF02492270. [CrossRef] [Google Scholar]
9. Sabah R. (2021). Simultaneous HPLC estimation of Amphetamine and Caffeine abuse drugs in Iraqi human addicts. Journal of Advanced Sciences and Engineering Technologies .; 2021 Dec. 13 ;4(1):25-31. Available from: https://isnra.net/ojs/index.php/jaset/article/view/13
10. Nagham Mahmood Aljamali. (2022). Spectral and Laboratory Diagnostics of Compounds. 1st Ed. Eliva Press SRL., ISBN: 9781636482118
11. Liu Z., Zhao S., Wang R. a., Guanghua Y. Separation of polyhydroxylflavonoids by packed-column supercritical fluid chromatography. J. Chromatogr. Sci. 1999;37:155–158. [Google Scholar]
12. Sun S.Y., Jiang W.G., Zhao Y.P. Comparison of aromatic and phenolic compounds in cherry wines with different cherry cultivars by HS-SPME-GC-MS and HPLC. Int. J. Food Sci. Technol. 2012;47:100–106. doi: 10.1111/j.1365-2621.2011.02812.x. [CrossRef] [Google Scholar]
13. N Mahmood A. (2018). Synthesis, Investigation, Chromatography, Thermal)- Behavior of (Five, Seven)- Membered Ring with Azo and Anil Derivatives . Pak. J. Biotechnol ., 15(1), 219-239
14. Agnieszka Wronka, Irena Malinowska ·, Wiesława Ferenc , Beata Cristovao., Chromatographic Study of Novel Heteronuclear Complexes with Schiff Base as Main Reagent., Chromatographia (2014) 77:1103–1112 ., DOI 10.1007/s10337-014-2664-2.
15. Ren, Jun; Yao, Peng; Chen, Jingjing; Jia, Lingyun (2014). "Salt-independent hydrophobic displacement chromatography for antibody purification using cyclodextrin as supermolecular displacer". Journal of Chromatography A. 1369: 98–104. doi:10.1016/j.chroma.2014.10.009 . PMID 25441076.

© STM Journals 2024. All Rights Reserved

16. N Mahmood A, Zainab Mohamed Farhan. (2023). Anticancer Study of Innovative Macrocyclic Formazan Compounds from Trimethoprim Drug ., Egyptian Journal of Chemistry ,2023 , 66, 1., T, Manish. "How does column chromatography work?". BrightMags. Archived from the original on 21 April 2017. Retrieved 7 April 2017.
17. N Mahmood A ."Separations of Samples Contents (Vitality, Environmental ,Chemical) By Instrumental & Laboratory Methods".,1 Edt. ,Evincepub Publishing, 2019., ISBN :978-93-88277-87-7 .
18. Müller, Tobias K.H; Franzreb, Matthias (2012). "Suitability of commercial hydrophobic interaction sorbents for temperature-controlled protein liquid chromatography under low salt conditions". Journal of Chromatography A. 1260: 88 -96. doi:10.1016/j.chroma .2012.08.052. PMID 22954746.
19. N Mahmood A. Survey on Methods of Preparation and Cyclization of Heterocycles. International Journal of Chemical and Molecular Engineering. 2020; 6(2): 10–27p.
20. Ren, Jun; Yao, Peng; Chen, Jingjing; Jia, Lingyun (2014). "Salt-independent hydrophobic displacement chromatography for antibody purification using cyclodextrin as supermolecular displacer". Journal of Chromatography A. 1369: 98–104. doi:10.1016/j.chroma.2014.10.009. PMID 25441076.
21. Prebihalo, Sarah E.; Berrier, Kelsey L.; Freye, Chris E.; Bahaghighat, H. Daniel; Moore, Nicholas R.; Pinkerton, David K.; Synovec, Robert E. (2018). "Multidimensional Gas Chromatography: Advances in Instrumentation, Chemometrics, and Applications". Analytical Chemistry. 90 (1): 505–532. doi:10.1021/acs.analchem.7b04226. ISSN 0003-2700.
22. Nagham M A.; Saher M Jawd.; Zainab M J.; Intisar, Obaid. Alfatlawi.; 2017. Inhibition activity of (Azo–acetyl acetone) on bacteria of mouth .,Research Journal of Pharmacy and Technology, 10(6):1683-1686, DOI: 10.5958/0974-360X.2017.00297.9
23. N Mahmood A.; S Mahmood Jawd.; Zainab M J.; Intisar, Obaid. Alfatlawi.; 2017. Inhibition activity of (Azo–acetyl acetone) on bacteria of mouth .,Research Journal of Pharmacy and Technology, 10(6):1683-1686, DOI: 10.5958/0974-360X.2017.00297.9
24. Hind Abdulhussein A., Samira Kazem H. (2023). Isolation and identification of Proteus mirabilis bacteria from different clinical sources and investigation of some of their virulence factors., Journal Alharf ., Issue August, No. 18, P: 148-160.
25. N Mahmood A. Synthesis and Chemical Identification of Macro Compounds of (Thiazol and Imidazol). Research J. Pharm. and Tech., 2015,8(1), 78-84., DOI: 10.5958/0974-360X.2015.00016.5.
26. Rusul Najm ., J Mohammed Hussein . (2023). Detection of Virulence Genes of Pathogenic Escherichia coli Isolated from Different Sources., Journal Alharf ., Issue August, No. 18, P: 130-138.
27. Stoll, Dwight R.; Carr, Peter W. (2017). "Two-Dimensional Liquid Chromatography: A State of the Art Tutorial". Analytical Chemistry. 89 (1): 519–531. doi:10.1021/acs.analchem.6b03506. ISSN 0003-2700.
28. Dhuha Raheem AA., Zainab Abd A M. (2023). Biosynthesis, characterization, Antioxidant and Antihemolysis activity of Silver nanoparticles (AgNPs) produced using Cynophyta Alga Extract (Spirulina platensis)., Journal Alharf., Issue. November, No. 19
29. Nagham M Aljamali. (2015). Review in Azo Compounds and its Biological Activity. Biochem Anal Biochem, 4, 169, doi:10.4172/2161-1009.1000169.
30. Tranchida, Peter Q.; Sciarrone, Danilo; Dugo, Paola; Mondello, Luigi (2012). "Heart-cutting multidimensional gas chromatography: A review of recent evolution, applications, and future prospects". Analytica Chimica Acta. A selection of papers presented at the 12th International Symposium on Extraction Technologies (ExTech 2010). 716: 66–75. doi:10.1016/j.aca.2011.12.015. ISSN 0003-2670.
31. Hawraa G H S , Zahraa S R N, Kawther K Abd-Alrudhe H , Afrah M A , Ban S Abd Alamer A , Suhair A k H , Zainab M A, Zainab A M. (2024). Review on Cancer and Its relationship to Medicinal Substances Found in Plants., Journal Alharf ., No. 20 ., February 2024

© STM Journals 2024. All Rights Reserved

32. Mhamd Abl, Abd Ali, N Mahmood A. Synthesis, Spectral, Bio Assay, Chromatographic - Studying of New Imidazole Reagents via Three Components Reaction. NeuroQuantology. 2021; 19(7):115-122, doi: 10.14704/nq.2021.19.7. NQ21092
33. Nemah Sahib MH, Kadhium AJ, Wannas FA, N Mahmood A. (2020). Novel Formazane Reagents and Reagents (Preparation, Spectral Characterization, Scanning Microscopy, Chromatographic Separation). Prensa Med Argent, Volume 106, 6, 303.
34. Hassan AK, Atiya MA, Luaibi IM. A Green Synthesis of Iron/Copper Nanoparticles as a Catalytic of Fenton-like Reactions for Removal of Orange G Dye. Baghdad Sci.J . 2022 Dec. 1 ;19(6):1249. Available from: https://bsj.uobaghdad.edu.iq/index.php/BSJ/article/view/6508
35. Singh NK, DSouza RN, Bibi NS, Fernández-Lahore M (2015). "Direct Capture of His6-Tagged Proteins Using Megaporous Cryogels Developed for Metal-Ion Affinity Chromatography". In Reichelt S (ed.). Affinity Chromatography. Methods in Molecular Biology. Vol. 1286. pp. 201–12. doi:10.1007/978-1-4939-2447-9_16
36. Stoll DR, Carr PW. (2017). "Two-Dimensional Liquid Chromatography: A State of the Art Tutorial". Analytical Chemistry. 89 (1): 519–531. doi:10.1021/acs.analchem.6b03506. PMID 27935671.
37. Tranchida PQ, Sciarrone D, Dugo P, Mondello L . (2012). "Heart-cutting multidimensional gas chromatography: a review of recent evolution, applications, and future prospects". Analytica Chimica Acta. A selection of papers presented at the 12th International Symposium on Extraction Technologies (ExTech 2010). 716: 66–75. Bibcode:2012AcAC..716...66T. doi:10.1016/j.aca.2011.12.015. PMID 22284880.
38. Berthod A, Maryutina T, Spivakov B, Shpigun O, Sutherland IA. (2009). "Countercurrent chromatography in analytical chemistry (IUPAC Technical Report)". Pure and Applied Chemistry. 81 (2): 355–387. doi:10.1351/PAC-REP-08-06-05. ISSN 1365-3075.
39. Jürgen Martens, Bhushan, R., Mieczysław Sajewicz, Teresa Kowalska J. Chromatogr. Sci. 2017, Vol. 55, 748–749. (doi:10.1093/chromsci/bmx031
40. Kulsing C, Nolvachai Y, Marriott PJ, Boysen RI, Matyska MT, Pesek JJ, Hearn MT .(2015). "Insights into the origin of the separation selectivity with silica hydride adsorbents". The Journal of Physical Chemistry B. 119 (7): 3063–9. doi:10.1021/jp5103753

© STM Journals 2024. All Rights Reserved