

## ORIGINAL ARTICLE



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# Designation of Macrocyclic Sulfazan and Triazan as Innovated Compounds with Their Estimation in Nano-Activities by the Scanning Microscope

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## Abstract

Sulfazan and Triazan Compounds were created for the first time as a class of organic chemistry in 2019 globally for the first time, then developed by researcher Dr. Nagham Aljamali in April 2021, while same researcher created and invented Sulfazan compounds as a nano-compounds, anti-bio compounds, and since the references are few on Sulfazan, so we have prepared a many of them and studied their applications in various fields to enrich this topic with references. A numeral of practical spectroscopic studies have been used to exhibit their chemical structures which provided strong evidences of their chemical structures through various technical devices like (FT IR-Spectra, <sup>1</sup>H.NMR-Spectra, Mass-Spectra), Melting points, other studies exemplified by evolution them as a Nano-compounds by Scanning Electron Microscopy (FESEM).

**Keywords:** *Cyclic Sulfazan, Triazan, Scanning Electron Microscopy (FESEM), Imine, Schiff base, Azo, Aldamine, Anil, Sulfazan.*

## Introduction

**Sulfazan compounds:** are the newest compounds in organic chemistry that were invented and developed by researcher Prof. Dr. Nagham Aljamali in 2019. The researcher has developed all methods<sup>(1, 2, 3)</sup> of their preparation, their properties and reactions, and studied their applications in several applied researches this is due to the references are a few on Sulfazan compounds<sup>(4-7)</sup>, for this reason the researcher Dr. Nagham carried out

many researches<sup>(8-10)</sup> in this field and carried out various studies to provide references on the methods of preparing and applying these newly invented compounds, so the researcher Dr. Nagham worked on several studies<sup>(1-8)</sup> of applications for these compounds, including microscopic scanning to prove that they are nano-compounds that act as drug carriers, anti-fungal, anti-bacterial, anti-tumors, and studies have been conducted for chromatographic separation of a sequences of them to study the effect of the active groups in these compounds<sup>(1, 3)</sup>.

**Triazan compounds:** are a newly invented compound in organic chemistry that were innovated and developed by researcher Prof. Dr. Nagham Aljamali in 2019. The researcher devised and innovated several procedures<sup>(1-3)</sup> to prepare it using several conditions and different catalysts according to the type of reactant of amine compounds in the reaction. The researcher studied many industrial

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applications and Bio-applications for these newer compounds to provide references<sup>(3-7)</sup> in this field., Cyclic Triazan Compounds have cyclic structure of (-N=N-N- in cyclic structure) according to type of amine derivative .

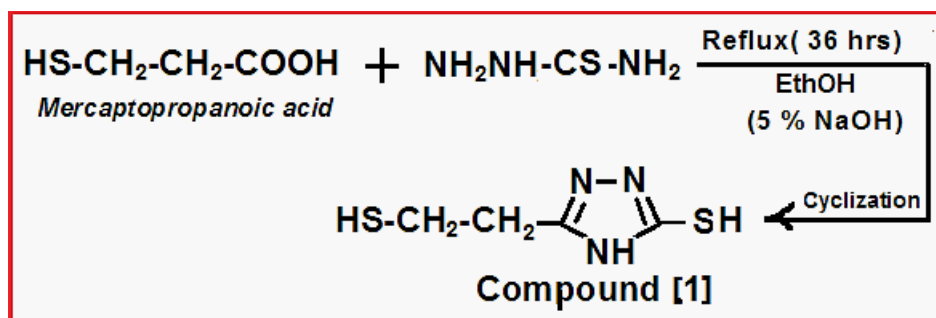
**Cyclic Sulfazan Compounds:** are Original-Invented compounds in organic chemistry field and are considered a new innovation by Dr. Nagham Aljamali in April 2021 when these compounds were prepared for the first time globally<sup>(1, 2)</sup>, and because their references and papers are few in this field for this reason the researcher Dr. Nagham Aljamali developed several procedures for preparation these compounds Macrocylic Sulfazan via using numerous conditions and different medium<sup>(3-7)</sup> like (Pyridine, Pipridine, graduated concentrations 5-7 % of Sodium hydroxide, Triethyl amine,...)<sup>(1, 2)</sup>, and linked them with heterocyclic compounds and other compounds like any heterocyclic compounds <sup>(11-20)</sup> with more than two hetero atoms to increase their effectiveness<sup>(21-30)</sup>, biological<sup>(31-40)</sup> and industrial applications<sup>(40-44)</sup>., Cyclic Sulfazan Compounds have cyclic structure of (-N=N-S- in cyclic structure) according to type of amine or sulfide compound.

**Instruments and Experimental Part:** All melting points were uncorrected and dignified on an electro-thermal apparatus (Switzerland) in an open capillary tube. FT.IR spectra were exhausted on Fourier transform infrared spectrometer (FT-IR) in( FT-IR- 3600) infrared spectrometer via employing KBr -Capsule technique., <sup>1</sup>H.NMR spectra were recorded in DMSO-d<sub>6</sub> as solvent using (TMS) as internal standard and chemical shifts are expressed as (δ ppm)., also Mass- Spectra for some of them other studies represented by evolution them as Nano-compounds by Scanning Electron Microscopy (FESEM).

### Experimental Approaches:

#### Preparation of Thiol-Triazole Derivative{1}

Mercaptopropanoic acid (0.01 mole) was reacted with thiosemicarbazide(0.01 mole) in (30 ml) absolute ethanol in basic medium (5 % NaOH) in rotation and refluxing step for (36 hrs), then cyclization step according to procedures<sup>(8, 9)</sup>, the product filtered, dried, recrystallized to yield derivative of Triazole thiol compound [1] by following literatures<sup>(8, 9)</sup>.



Scheme.1: Synthesis of Thiol-Triazole Derivative [1]

#### Preparation of Invented Sulfazan Compounds{2}

The derivative of Triazole thiol Compound [1] reacted (0.01 mole) with (0.02 mole) from diazo salt of anthranilic acid in Pipridine through three steps in basic medium through rotation steps to formation Invented Sulfazan compound after (15 hrs), the product filtered, dried, washed by distilled water, recrystallized to yield Invented Sulfazan Compound [2] by following literatures<sup>(1-3)</sup>.

#### Preparation of MercaptoTriazole- Sulfazan Compounds{3}

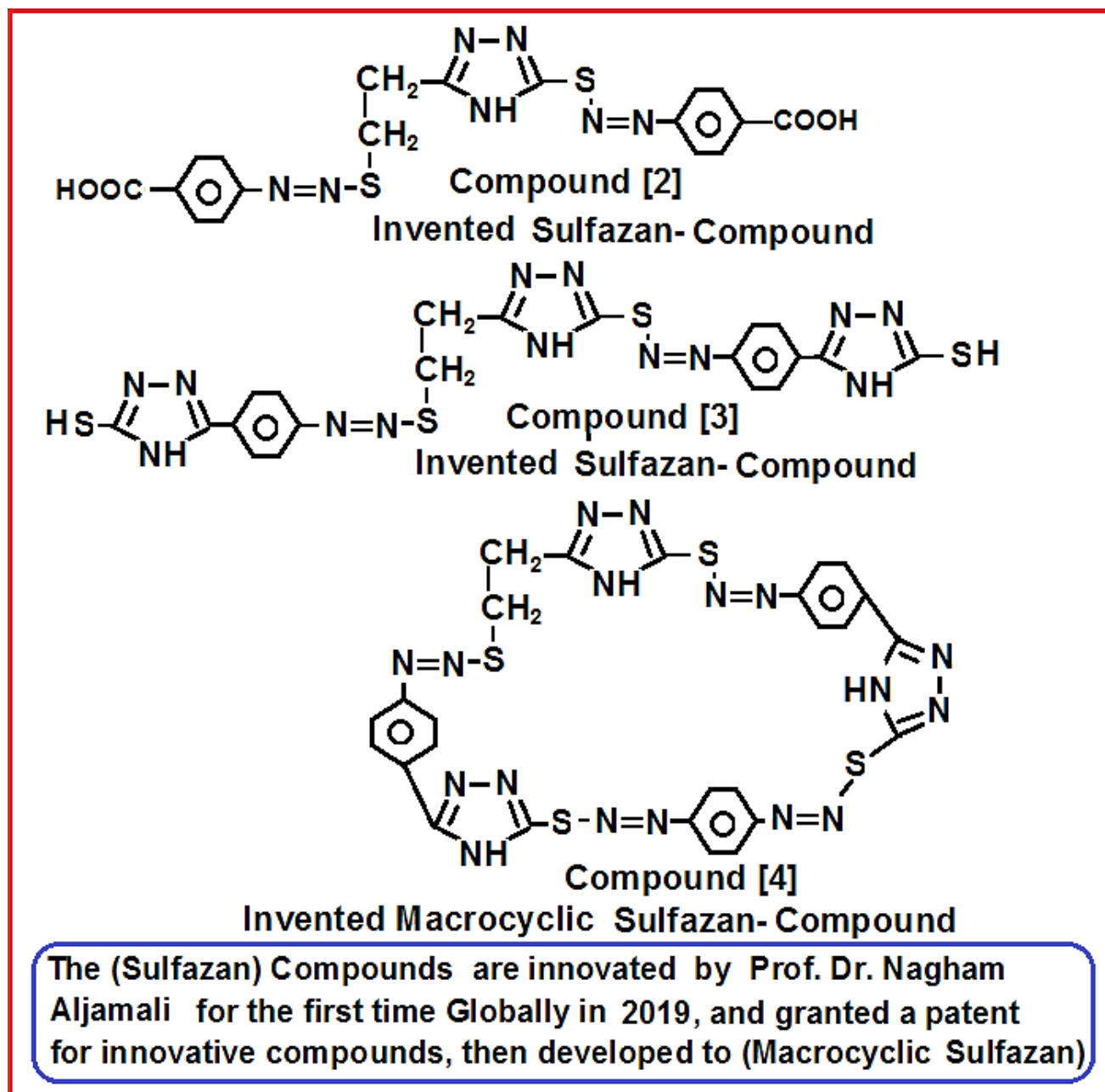
Invented Sulfazan compound [2] (0.01 mole) was reacted with thiosemicarbazide(0.01 mole) in (30 ml)

absolute ethanol in basic medium (6 % NaOH) in rotation and refluxing step for (48 hrs), then cyclization step according to procedures<sup>(1-3)</sup>, the product filtered, dried, recrystallized to yield derivative of bis-(mercaptoTriazole-Sulfazan) Compound [3] by following Invented Methods in literatures<sup>(1-3)</sup>.

#### Preparation of Invented Macrocylic Sulfazan Compounds{4}

derivative of bis-(mercaptoTriazole-Sulfazan) Compound [3] reacted (0.01 mole) with (0.01 mole) from diazo salt of phenyl diamine in Pyridine through three steps in basic medium through rotation steps to formation Invented Macrocylic Sulfazan compound after (30 hrs),

the product filtered, dried, washed by distilled water to yield Invented Macrocylic Sulfazan Compound [4] by following Invented Methods in literatures<sup>(1-3)</sup>.

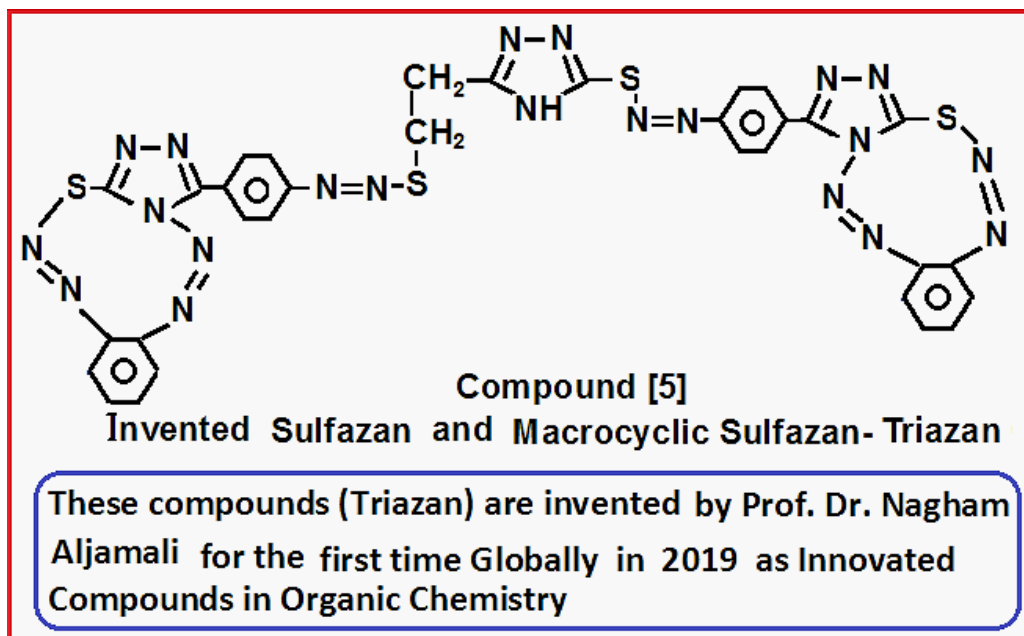


Scheme 2: Creation of Linear Sulfazan and Macrocylic Sulfazan Compounds {2, 3, 4 }

#### Creation of Inventive Macrocylic Triazan Compounds { 5 }

Compound [3] was reacted (0.01 mole) in presence<sup>(1,2,9)</sup> of (Pipyridine) with (0.02 mole) of diazo salt of o-phenyl diamine via many steps in basic medium

to formation Invented Macrocylic Triazan after (52 hrs), the product filtered, dried, washed by distilled water to yield Invented Macrocylic Triazan Compound [5] linked with Sulfazan group in same Macrocycle, by following Invented Methods in literatures<sup>(1-3)</sup>.



Scheme 3: Creation of Invented Macrocyclic Sulfazan and Triazan Compounds{5}

## Results and Discussion

In the recent scientific paper, a number of Invented Macrocyclic Sulfazan and Triazan Compounds have been created in same procedure that followed and invented<sup>(1-3)</sup> by Dr. Nagham in year 2019, then several studies were carried out to improve these innovative compounds by the using of spectral identification like: <sup>1</sup>H.NMR spectra, FT.IR- Spectra, Mass- Spectra., other studies represented by (Melting points, other studies performed with evolution them as Nano-compounds by Scanning Electron<sup>(34)</sup> Microscopy (FESEM), all the results are revealed in Tables and figures:

### Spectral Evidences:

**FT.IR- Spectral Evidence of Invented Macrocyclic Sulfazan and Triazan Compounds:** The first evidence of innovated Sulfazan and Triazan compounds by shifting of frequencies of Azo group (-N=N) in starting compounds that was at (1455, 1490)  $\text{Cm}^{-1}$  to (1442, 1485, 1505)  $\text{Cm}^{-1}$  in New Sulfazan due to Sulfazan group (-N=N-S) and Triazan Compound for triazan group (-N=N=N-) due to shifting and splitting of the frequency in the band of the infrared spectrum into three bands in prepared compound (Sulfazan and Triazan Compounds) instead of two bands (starting materials: Azo compound) in all formatted Sulfazan and Triazan Compounds., while disappearance

the band of thiol group (SH) in Compounds [1, 3] and appearance new bands in compounds [2, 4, 5] at (1205, 1214)  $\text{cm}^{-1}$  due to sulfide in Sulfazan group (-S=N=N-), also appearance of three bands due to partitions of triazan group of Macrocyclic of Triazan compound (N=N=N-) are three bands (1425, 1461, 1496)  $\text{Cm}^{-1}$  for (-N=N=N-) in compound {5}, also appearance band at (1313)  $\text{Cm}^{-1}$  due to (-N=N-) in Triazan compound [5]., all frequencies clarified and explained according to references<sup>(1,33)</sup>.

### <sup>1</sup>H.NMR- Spectral Evidence of Invented Macrocyclic Sulfazan and Triazan Compounds:

The second evidence of innovated compounds by disappearance of peak for thiol group (SH) in starting compound (Mercapto compounds) that was at  $\delta$  (4.02) in Compounds {1} and at  $\delta$  (4.14) in Compound {3} respectively in (starting compound) due to formation of (R-S-N=N) Sulfazan in compounds {2, 4, 5}., Also disappeared peak that was at  $\delta$  (5.18) due to removing the proton of amine in Triazole ring(NH) due to formation (-N=N=N- in cycle structure) of Triazan compound [5], all peaks explained according to reference<sup>(33)</sup>.

### Mass- Spectral Evidence of Invented Macrocyclic Sulfazan and Triazan Compounds :

The third evidence of innovated compounds via partition of invented cyclic compounds via appearance of fragments in spectra in figures (1, 2, 3).

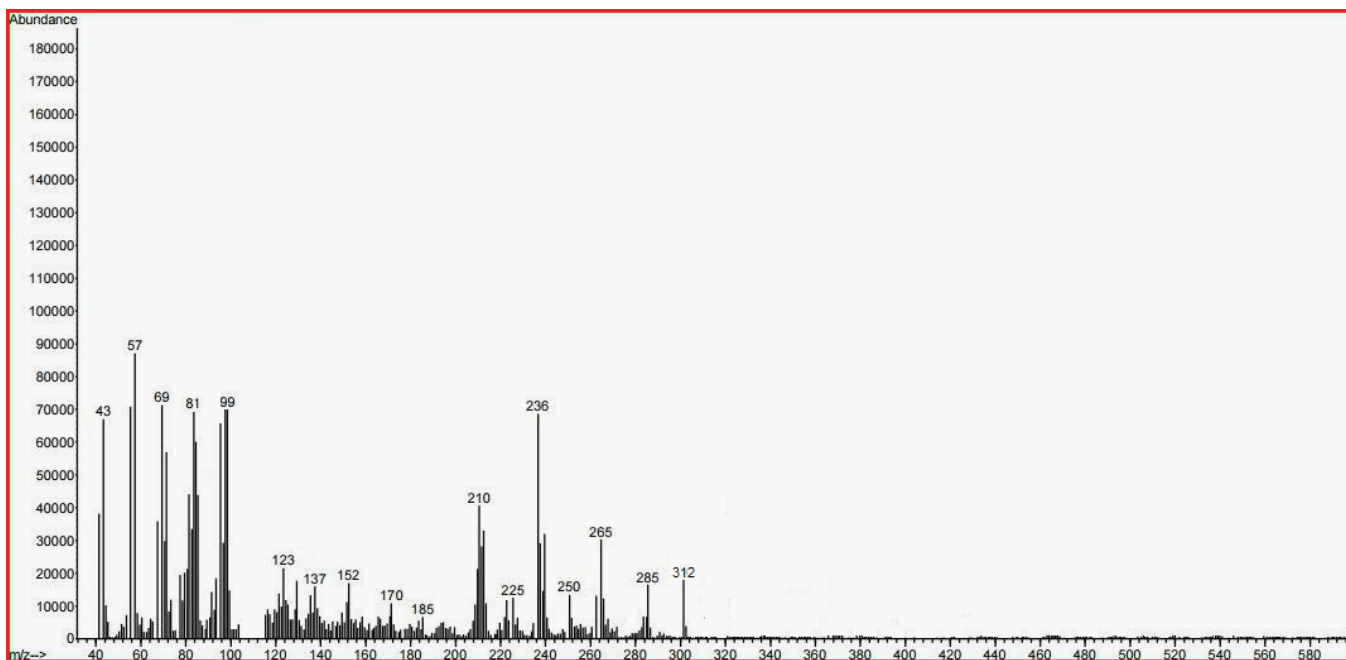


Fig. (1): Mass-Spectrum of Invented Macrocyclic Sulfazan Compound {2}

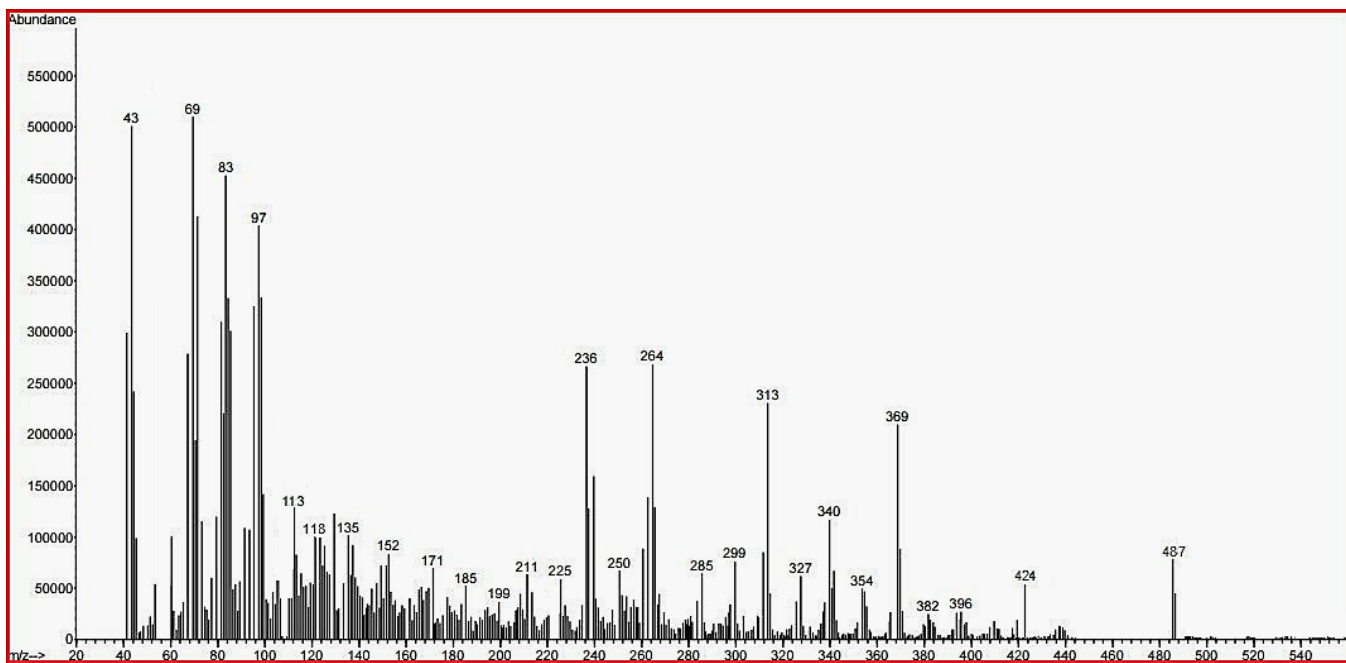


Fig. (2): Mass-Spectrum of Invented Macrocyclic Sulfazan Compound {4}

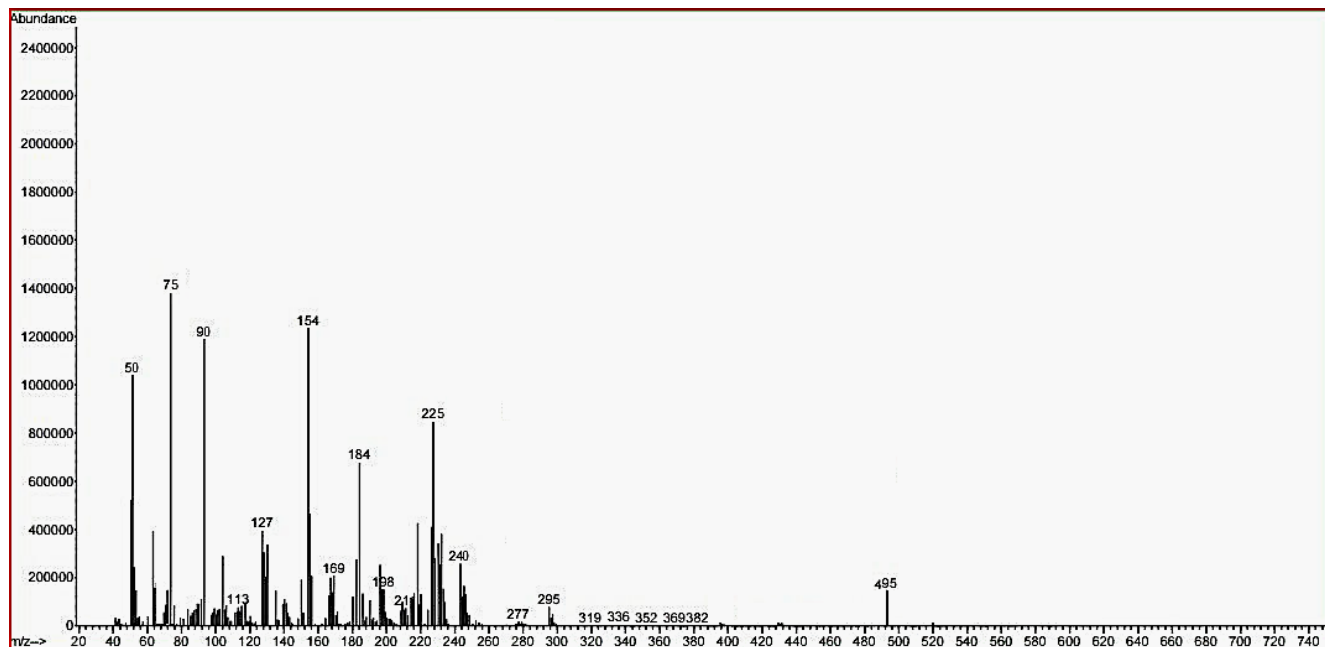


Fig. (3): Mass-Spectrum of Invented Macrocylic Triazan Compound{5}

**Other Characterization:**

All Invented Macrocylic Sulfazan Compounds and Triazan derivatives were studied to collect all the chemical and physical properties, in table(1) :

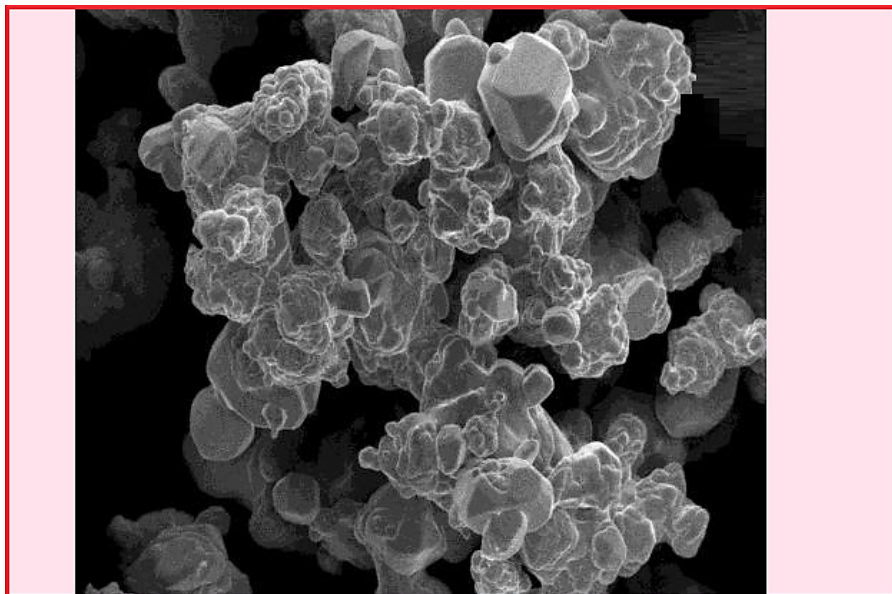
**Table 1: Other categorization of Invented Macrocylic Sulfazan and Triazan Compounds**

Innovated Comps.	P%	Color	M.P C°	Rf	Solvents (TLC)
Innovated Comp. {1}	68	Deep Yellow	162	0.62	Ethanol : Hexane
Innovated Comp. {2}	84	Bill Orange	198	0.68	Ethanol : Hexane
Innovated Comp. {3}	70	Deep Yellow	212	0.58	Ethanol : Hexane
Innovated Comp. {4}	88	Orange	226	0.64	Ethanol : Hexane
Innovated Comp. {5}	86	Orange	230	0.68	Ethanol : Hexane

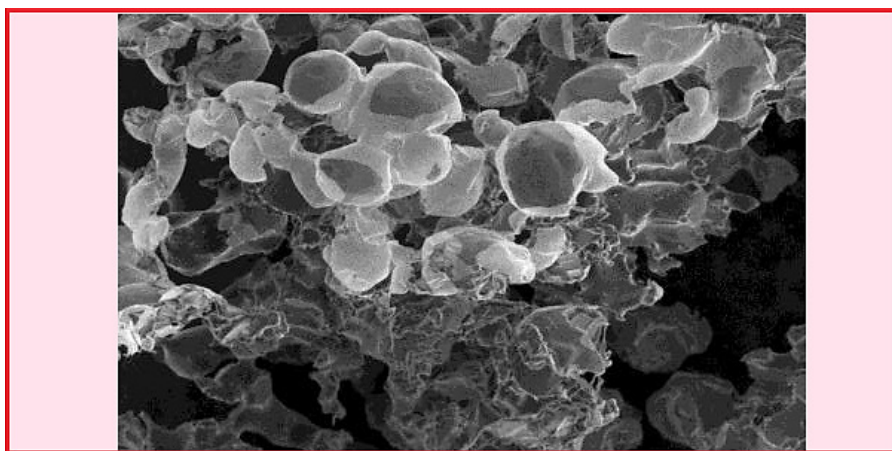
**Scanning Electron Microscopy (FESEM)<sup>(34)</sup>:**

Scanning Electron Microscopy (FESEM) of the Invented Macrocylic Sulfazan compounds and Triazan compound (for morphological properties) that discovered in this research that they have a spherical shape and have granular sizes within the nano-scale they have an average size of (40. 18, 42. 04, 48. 78) nanometers for Macrocylic

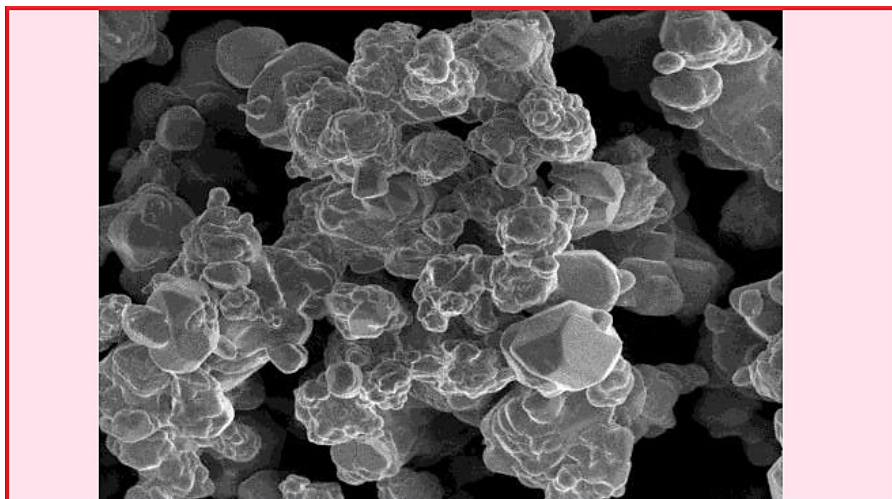
Sulfazan and Triazan Compounds [2, 4, 5] respectively by following procedure<sup>(34)</sup>, so the surface area increases and this characteristic makes it eligible for medical uses because it has a small granular size, spherical shape within the nano-scale that is used in medical fields as a treatment for many types of cancers as well as in the industrial field, figures (4, 5, 6):



**Fig. (4): Scanning Electron Microscopy of Invented Linear Sulfazan [2]**



**Fig. (5): Scanning Electron Microscopy of Invented Macrocyclic Sulfazan [4]**



**Fig. (6): Scanning Electron Microscopy of Invented Cyclic Triazan [5]**

## Conclusions

All Spectral Techniques of Invented Macrocyclic Sulfazan and Macrocyclic Triazan compounds appeared good evidences for their structures via various spectral methods, also some of them studied as a nano-compounds that gave good data by using Scanning Electron Microscopy (FESEM).

**Ethical Clearance:** Ethics committee refer that there is no plagiarism and there is no mistakes or wrong results or plagiarism in this work.

**Conflict Of Interest:** The authors declare that there is no conflict of interest.

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