MONITORING OF COUNTERFEIT ABAMECTIN PESTICIDE PRODUCTS IN EGYPT

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ABSTRACT

The monitoring of the counterfeit situation of a pesticide widely used in Egypt (abamectin) was done through the present study as seven samples of Abamectin formulations were collected from the Egyptian market. Packaging and labels were checked and analyzed by HPLC to determine the active ingredient content present in these samples. GC-MS and FTIR were used for additional analysis and detection of other active ingredients in the formulation.

Counterfeit pesticides were studied in Egypt by doing questionnaires for all workers in the pesticide system (farmer - trader - researchers in the pesticide field) and the results of these forms were analyzed to show the most dominant pesticides in Egypt, in addition to random purchase of pesticides from the Egyptian market and the most number of samples is from (abamectin pesticide) (7 samples). To obtain this number of abamectin formulations, 58 different pesticide formulations (16 different active ingredients) were collected from the Egyptian markets, and the share of abamectin was 7 samples (one active substance), representing 12.1% of the total tested samples.

The obtained results showed that Examination of Packaging and indicated that three samples were not registered through Egyptian Agricultural Pesticides Committee thus representing 42.86% of the total tested samples. The non-registered products are Komaktin Gold Plus, Super Vertimic and Abamectine Strela. Three samples have registration number as other formulations already registered in Ministry of Agriculture which are Abamectin Super, Farmactine and Abamectin power. The last product (Tinam) has the correct registration number (no.1391) and it is the same number used to register it in Ministry of agriculture.

The percentage of the active ingredient in 5 samples was less than the acceptable limits, The samples were (Abamectin super, Komaktin Gold Plus, Abamectin Power, Super vertimic and Abamectine strela) active ingredient content was 1.416, 0.64, 1.01, 0.2 and 0.12% respectively. One sample (Farmactine) didn’t contain Abamectin, and the sample (Tinam) was within the acceptable limits (1.53%).

GC-MS used to determine the presence of any other materials or active ingredients within the samples. The result showed that Abamectin Super sample contains (Lambda cyhalothrin at R.t. 29.512 min), Farmactine sample contains (Fenpropathrin at R.t. 28.634 min), Abamectin Power sample contains (Diazinon at R.t. 16.088 min and Cypermethrin at R.t. 26.554 min) and Super Vertimic sample contains (Fenpropadrin at R.t. 23.916 min and Lambda cyhalothrin at R.t. 34.85 min). Three products Komaktin Gold Plus, Tinam and Abamectine Strela didn’t contain other active ingredients.

The physical properties of the studied samples before and after storage were determined through emulsion characteristic test and four samples (Abamectin super, Farmactine, Abamectin Power and Tinam) showed good emulsion characteristics before and after storage. Super vertimic and Abamectine strela samples showed poor emulsification characteristics before and after storage. Komaktin Gold Plus showed good emulsification before storage but poor emulsion characteristics after storage.

Keywords: Counterfeit Pesticides; Illegal Pesticides; Abamectin; Mass Spectroscopy; IR; Physical properties
INTRODUCTION

The Agriculture Industry, which is the backbone of our economy, is facing multiple threats from the rise in counterfeit pesticides. There is rapid worldwide growth in counterfeit and illegally traded pesticides. These fake products are manufactured and sold by criminal groups. Based on the specificities of nations, the scope and scale of fake pesticides differ from store to store. Depending on the circumstances of the countries, Counterfeit plant protection products can cause losses of crops and threaten public health, food trade and the environment.

In 2018, the global chemicals market for crop protection was worth USD 57.5 billion. It is projected to reach USD 68.82 billion by 2025, (Agrochemicals Market by Type – Global Trends & Forecast to 2025) and is expected to increase by 4.7% during the forecast period (2019-2024). (Crop Protection Chemicals Market)

According to estimated data published by the European Crop Protection Association (ECPA, 2012), illegal and counterfeit pesticides account for over 10% of the worldwide market share of plant protection products. This percentage of market share in developing countries is projected to reach 20-30 percent (Karasali et al. 2014). Based on ECPA statistics, 8-10 percent of plant protection products on the European market are estimated to be counterfeit. Concerning the Polish market, the scale is estimated at 10–15%. The main reason why this practice is widespread is financial benefits. Fake and illegal pesticides that contain materials that could pose a threat to the crops, environment, animals, and people.

Counterfeiting of pesticides and crop protection products has been a troublesome issue for the pesticide industry. Isolated instances are usually identified when a crop fails or the product does not work, and have been dealt with by individual companies, more recently with the growth of chemical manufacturing capability in countries such as India and China.

Pesticides are of great importance, necessity due to their use in agriculture and public health, therefore it is necessary to ensure the quality of these pesticides and their conformity with the standard specifications, especially since their active ingredients are no longer manufactured by their original producer once their patent period of 20 years has expired.

During the summer of 1976, thousands of cases of poisoning resulting from the use of malathion for malaria vector control were reported in Pakistan, that resulted in at least five deaths. Although these poisoning cases were primarily attributable to poor safety practices and hygiene, there was evidence that increased toxicity of poor-quality malathion also contributed to the poisonings. (Aldridge et al 1979).

The poor quality of malathion used in Pakistan had an S-methyl isomer content of 3.1 % as well as significant amounts of the O,O,S-trimethylphosphorothioate and O,S,S-trimethyl phosphorodithioate impurities. Thus, the effect of such impurities may have had undesirable effects on human.

The first case of illegal pesticides in Europe occurred in Spain in the year 2000, when authorities uncovered unregistered pesticides without labels being imported from China (European Commission DG Health and Food Safety, 2015).

The nature and extent of counterfeit pesticides and illegal trade varies per market and can originate from many different sources in many different forms. The three main areas of illegal activity are:

1. Fakes: containing something from water or talc to diluted and obsolete or outdated products, including forbidden or banned materials Many fakes may provide a level of biological control, as they mostly contain an illegal and untested copy of the proprietary active substance.
2. Counterfeits: Advanced copies of branded products usually with high quality labeling and packaging. Most will contain a copy of the original active substance however; its biological efficacy is often less owing to high levels impurities of manufacturing and process by-products.
3. Illegal parallel imports: Parallel traded products substituted with illegal generic copies, repackaged and sold as original products. The repackaging puts the products at risk, allowing for contamination and the use of unacceptable packaging leads to a bad product that may cause harm to crops and pose risks to consumers. (Frederick M. Fishel, 2008).

Bad effects of illegal trade and counterfeit pesticides:

1. The health of consumers and farmers is at risk.
2. Environmental damage.
3. Economic damage to farmers.
4. Economic damage to governments.
5. Economic damage to the plant protection industry.
All illicit products present a host of challenges to enforcement of trade regulations and controls (including taxation avoidance), industrial regulation and operations, farming operations, and consumer behavior and safety. Illicit goods also raise fundamental questions about legal and economic responsibility, social harm, and how illicit products and precursors are insinuated into legitimate distribution and supply chains. As mentioned, deleterious impacts of illicit pesticides are numerous and include damage to human and animal health, economic development and trade, agricultural production, natural resources and water supplies, including indigenous plants and wildlife.

Fighting counterfeit pesticides is a very complex task. We see there are more and more regulations related to pesticide use, but less and less attention towards implementing these regulations. Pesticide producers are dedicating human and financial resources to fighting illegal trade and counterfeits. But they cannot succeed alone. Those responsible, and affected, need to lead governments, sellers and farmers. There is an immediate need for increased focus and improved human and financial resources.

The present work was directed to monitor the counterfeit situation of abamectin pesticide in Egypt.

MATERIAL AND METHODS

1. Pesticides used

The current work has been carried out to monitor the counterfeit situation of the abamectin formulations used in the agrochemicals market in Egypt. This was done through analyzing their active ingredient content by HPLC. GC-MS and FTIR were used to check for other substances present in the products and finally, the physical properties of these samples was checked before and after storage using emulsion characteristic test.

In addition to this work, pesticide packages and labels were checked to monitor the position of counterfeit abamectin in Egypt.

Data on the active ingredient used for this work can be found in (Table 1) below.

2. Sampling

Seven pesticide formulations of emulsifiable concentrates of Abamectin were collected from several areas in the governorates of Cairo and Giza. Their trade names and concentrations on the label can be found in (Table 2) below.

3. Quantitative analysis of active ingredients of the investigated pesticide formulations

3.1. Standard preparations of the tested pesticides.

Standard solutions were prepared in methanol, at 400 ppm for abamectin pesticides.

3.2. Sample preparations of the tested pesticide formulations

Weight of each tested formulation (Table 3) was placed into 25 ml volumetric flask and completed to volume with methanol and shaken to dissolve the sample.

4. Determination of active ingredients content of the tested pesticides by High Performance Liquid Chromatography

HPLC equipment (Agilent technologies 1260 Infinity) was used with UV detector. The column Eslips Plus C18, di.5 Mn and Len. 4,6 * 2.5 mm. The wavelength detector at 235 nm. The mobile phase was a mixture of acetonitril: methanol (70: 30). The flow rate was 1.3 ml/ min.

5. Determination of other materials present in the formulation

5.1. Gas Chromatography- Mass Spectroscopy

A stock solution (4000 ppm) of formulation samples was prepared in (25 ml) methanol. The GC-MS analysis was performed with an Agilent 7890 B, gas chromatograph equipped with a mass spectrometric detector (MSD) model Agilent 5977 A. A fused silica capillary column (30m x 0.25 mm HP-5-0.25 micron -60 to 325°C) was used. Samples were injected under the following conditions:

Helium as carrier gas at a flow rate of 1.0 ml/min, pulsed split mode, split ratio (10:1), split flow 10 ml/min. The solvent delay was 4 min and 1µl volume was injected. Column temperature was maintained at 50°C, for 0.5 min then programmed at 10°C/min to 190°C followed by 10°C/min ramp to 210°C for 1 min followed by 10°C/min ramp to 300°C and held for 2 min. Total analysis time was 29.5 min. Injector port temperature was set at 280°C.

Wiley mass spectral data base was used in the identification of the separated peaks.
Table 1. Abamectin data - Pesticide Manual (2003)

| Common name          | Abamectin                  |
|----------------------|----------------------------|
| Chemical class       | Avermectin                 |
| IUPAC name           | mixture with \((10E,14E,16E,22Z)-(1R,4S,5'S,6S,6'R,8R,12S,13S,20R,21R,24S)-21,24-dihydroxy-6'-isopropyl-5',11,13,22-tetramethyl-2-oxo-3,7,19-trioxatetracyclo[15.6.1.14.8.020,24]pentacosa-10,14,16,22-tetraene-6-spiro-2'-(5,6'-dihydro-2'H-pyran)-12-yl\) 2,6-dideoxy-4-O-(2,6-dideoxy-3-O-methyl-L-arabino-hexopyranosyl)-3-O-methyl-\(\square\)-L-arabino-hexopyranoside |
| Molecular formula    | \(\text{C}_{48}\text{H}_{72}\text{O}_{14}\) |
| Chemical structure   | [Chemical structure diagram] |
| Molecular weight     | 873.1                      |
| Activity             | Control of motile stages of mites, leaf miners, suckers, Colorado beetles, etc. on ornamentals, cotton, citrus fruit, pome fruit, nut crops, vegetables, potatoes, and other crops. Application rates are 5.6 to 28 g/ha for mite control, 11 to 22 g/ha for control of leaf miners. Also used for control of fire ants. |

Table 2. Products collected from the market for testing.

| Trade Name                  | Active Ingredient | Suggested Abamectin content on the label |
|-----------------------------|-------------------|----------------------------------------|
| Abamectin Super EC          | Abamectin         | 1.8%                                   |
| Farmactine EC)              | Abamectin         | 1.8%                                   |
| Komaktin Gold Plus EC       | Abamectin & P.M.O | & 5%                                   |
| Abamectin Power EC          | Abamectin         | 1.8%                                   |
| Super Vertimic EC           | Abamectin         | 1.8%                                   |
| Tinam EC                    | Abamectin         | 1.8%                                   |
| Abamectine Strela EC        | Abamectin         | 1.8%                                   |
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Table 3. Weights for each sample prepared for testing

| Trade Name                  | Weight (gm)/25 ml |
|-----------------------------|-------------------|
| Abamectin super (1.8% EC)   | 0.510             |
| Farmactine (1.8% EC)        | 0.490             |
| Komaktin Gold Plus (6.8% EC)| 0.560             |
| Abamectin Power (1.8% EC)   | 0.510             |
| Super Vertimic (1.8% EC)    | 0.290             |
| Tinam (1.8% EC)             | 0.497             |
| Abamectine Strela (1.8% EC) | 0.465             |

5.2. Infrared spectroscopy

The samples were analyzed using Fourier transform infrared (FTIR) model (AVATAR 330) FT-IR Thermo Nicolet. Samples were examined between salt plates usually without a spacer. Pressing a sample between flat plates produces a film of 0.01 mm or less in thickness, the plates were held together by capillarity. Samples of 1-10 mg were required to produce a satisfactory spectrum.

IR spectra were recorded (KBr) by a pye Unicam SP-1000 Spectrophotometer (υ max in cm⁻¹). According to Spectrometric Identification of Organic Compounds, fifth edition, infrared spectrometry, chapter three, 1991-a).

6. Determination of emulsion characteristics of the investigated formulations before and after accelerated storage test

6.1. Accelerated storage procedure (CIPAC MT 46.3).

The formulations were stored in a glass bottle in an oven at 54 ± 2°C for 14 days according to CIPAC MT 46.3 and the emulsification test was performed on the samples before and after storage to examine its physical properties.

6.2. Preparation of standard water

CIPAC MT 18.1 (1995), MT 18.3 Non-CIPAC Standard Waters, 18.3.1 WHO Standard Hard Water, (342 ppm hardness) were used in all tests of physical properties.

Calcium chloride CaCl₂ (0.304 g) and magnesium chloride MgCl₂ 6 H₂O (0.139 g) were dissolved in distilled water and made up to 1000 ml.

6.3. Emulsion stability evaluation for abamectin formulations (EC)

The test was carried out according to CIPAC MT 36.3 (1995) where 5 ml of the formulation was added to 95 ml of standard water (in cylinder 100 ml), the cylinder was stoppered then inverted once and left to stand for 30 seconds. Then the cylinder was inverted 10 times and left to stand at 30°C ± 2°C for 30 min in a water bath. Any separated materials, volume of free oil, froth, cream, or solid matter present, if any, were recorded at the end of the 30 min period.

RESULTS AND DISCUSSION

Abamectin formulations of seven samples were collected from seven Egyptian markets then analyzed by HPLC to determine their active ingredient content. GC-MS and FTIR were used to determine the presence of other ingredients within the formulation and the physical properties of the studied products were also considered before and after storage using emulsion characteristics test.

1. Abamectin formulation labels

Table (4) shows the recorded data on labels of seven Abamectin formulations collected from different markets of Cairo and Giza. Examination of tabulated data indicated that three samples were not registered through Egyptian Agricultural Pesticides Committee from seven samples are the total tested samples. The non-registered products are Komaktin Gold Plus, Super Vertimic and Abamectine Strela.

Three samples have registration number as other formulations already registered in Ministry of Agriculture which are Abamectin Super, Farmactine and Abamectin power. The last product (Tinam) has the correct registration number (no.1391) and it is the same number used to register it in Ministry of agriculture.

2. Physical Properties of Abamectin formulations

Emulsion test was performed before and after storage of the abamectin formulations. Samples were stored in a glass bottle in an oven at 54±2°C for 14 days.
Table 4. Data from product labels.

| Item                | Abamectin super | Farmactine | Komaktin Gold Plus | Abamectin power | Super vertimic | Tinam | Abamectine strela |
|---------------------|-----------------|------------|---------------------|-----------------|----------------|-------|-------------------|
| Registration no     | 1774            | 466        |                     |                 | 1020           |       |                   |
| Common name         | Abamectin       | Abamectin  | Abamectin 1.8% P.M.O 5% | Abamectin      | Abamectin      | Abamectin | Abamectin       |
| % a.i.              | 1.8%            | 1.8%       | 6.8%                | 1.8%            | 3.6%           | 1.8% | 1.8%              |
| Formulation type    | EC              | EC         | EC                  | EC              | EC             | EC    | EC                |
| Toxicity class      | High lb         | -----      | -----               | -----           | -----          | ------| ------            |
| Company             | Chimac - agriphar | Hand limited | Power Chemicals Germany- Frankfurt | ----- | The King Zife Biochemical Co., Ltd. - China. | The King Zife Biochemical Co., Ltd. - China. | Petrochem- Dubai Jabel Ali United Arab Emirates |
| Imported company    | Modern chemicals industries co | ----- | ----- | Aspire for protection and development of agricultural projects | Harvest Group Chemical | Pharmaciotica Chemicals and Pharmaceuticals. | Future Green |

--- : not available
The emulsification test before and after storage conforms to specifications.

Fig. 1. Emulsion test before and after storage of Abamectin super.

The emulsification test before conforms to specifications but after storage doesn’t conform to specifications (Sample color changed) (Volume of Sediment is less than 2 ml)

Fig. 3. Emulsion test before and after storage of Komaktin Gold Plus.

The emulsification test before and after storage conforms to specifications.

Fig. 4. Emulsion test before and after storage of Abamectin power.
The emulsification test before and after storage doesn’t conform to specifications (Volume of Sediment/cream layer is more than 2 ml)

**Fig. 5.** Emulsion test before and after storage of Super vertimic.

The emulsification test before and after storage conforms to specifications

**Fig. 6.** Emulsion test before and after storage of Tinam.

The emulsification test before and after storage doesn’t conform to the specifications (Volume of Sediment/cream layer is more than 2 ml)

**Fig. 7.** Emulsion test before and after storage of Abamectine strela
The results indicated that 4 samples (Abamectin super, Farmactine, Abamectin Power and Tinam) showed good emulsification characteristics before and after storage. Super vertimic and Abamectine strela samples showed poor emulsification characteristics before and after storage. Komaktin Gold Plus showed good emulsification before storage but poor emulsion characteristics after storage.

3. Determination of active ingredients content of Abamectin formulations by HPLC.

The percentage of the active ingredient in 5 samples was less than the acceptable limits (+/-15%). The samples (Abamectin super, Komaktin Gold Plus, Abamectin Power, Super vertimic and Abamectine strela) active ingredient content was 1.416, 0.64, 1.01, 0.2 and 0.12% respectively. One sample (Farmactine) didn’t contain Abamectin, and the sample (Tinam) was within the acceptable limits. Results can be summarized in the Table 5 below:

| Trade Name               | Assay (% active ingredient) |
|--------------------------|------------------------------|
| Abamectin Super (1.8%)   | 1.416                        |
| Komaktin Gold Plus (1.8%)| 0.64                         |
| Abamectin Power (1.8%)   | 1.01                         |
| Super Vertimic (1.8%)    | 0.2                          |
| Abamectine Strela (1.8%) | 0.12                         |
| Farmactine (1.8%)        | 0                            |
| Tinam (1.8%)             | 1.53                         |

4. Determination of other materials present in the samples

4.1. GC-MS Analysis

Samples were analyzed by GC-MS to determine the presence of any other materials or active ingredients within the samples.

Abamectin Super sample contains (Lambda cyhalothrin at R.t. 29.512 min). Farmactine sample contains (Fenpropathrin at R.t. 28.634 min), Abamectin Power sample contains (Diazinon at R.t. 16.088 min and Cypermethrin at R.t. 26.554 min) and Super Vertimic sample contains (Fenpropethrin at R.t. 23.916 min and Lambda cyhalothrin at R.t. 34.85 min).

Three products Komaktin Gold Plus, Tinam and Abamectine Strela didn’t contain other active ingredients.

4.2. Identification of Abamectin by FTIR

FTIR analysis of Abamectin formulations was performed and the characteristic peaks were tentatively identified in the following Figs. (16, 17, 18, 19, 20, 21 and 22).

The different functional group revealed the following range wave number for sample as indicated in Table 6. It was noted that certain parent function groups are in range in all products, while some other bands were appeared such as (C≡N stretching) in samples Abamectin super, Farmactine and Abamectin Power. In samples Abamectin super and Super Vertimic (C-F) appeared at 1387 and 1377 respectively.

Counterfeiting has been reported in the form of absence of the active ingredient, wrong active ingredient. Active ingredient, fake packaging and contamination with unexpected substances (Karasali, 2015). The result showed that, the absence of the active ingredient in sample Farmactine. The insufficient active ingredient in samples Komaktin Gold Plus, Abamectin Power, Super vertimic and Abamectine strela. The packaging in samples Abamectin super, Farmactine, Komaktin Gold Plus, Abamectin power, Super vertimic and Abamectine strela. The contamination with unexpected substances in samples Abamectin super, Farmactine, Abamectin power and Super vertimic.
Fig. 8. HPLC Chromatogram of Abamectin St.

Fig. 9. HPLC Chromatogram of Abamectin super.

Fig. 10. HPLC Chromatogram of Farmactine.

Fig. 11. HPLC Chromatogram of Komaktin Gold Plus.
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Fig. 12. HPLC Chromatogram of Abamectin Power.

Fig. 13. HPLC Chromatogram of Super vertimic.

Fig. 14. HPLC Chromatogram of Tinam.

Fig. 15. HPLC Chromatogram of Abamectine strela.
Fig. 16. FTIR spectrum of Abamectin super

Fig. 17. FTIR spectrum of Farmactine.

Fig. 18. FTIR spectrum of Komaktin Gold Plus

Fig. 19. FTIR spectrum of Abamectin power

Fig. 20. FTIR spectrum of Super vertimic

Fig. 21. FTIR spectrum of Tinam

Fig. 22. FTIR spectrum of Abamectine strela
Table 6. IR absorbance of functional groups of abamectin formulations

| Type of bond (abamectin) | Abamectin super | Komaktin Gold Plus | Abamectin power | Super vertimic | Tinam | Abamectine strela |
|--------------------------|-----------------|-------------------|-----------------|----------------|-------|-------------------|
| O-H stretching           | 3435            | 3427              | 3423            | 3447           | 3451  | 3430              | 3447 |
| -C-H (aromatic)          | 2925            | 2955- 2924        | 2925            | 3018- 2962     | 3018- 2956 | 3019- 2924        | 2955- 2924 |
| -C-H (aliphatic)         | 2872            | 2854              | 2856            | 2924- 2872     | 2924- 2854 | 2871              | 2854 |
| C=O (carbonyl)           | 1672- 1610      | 1741- 1612        | 1740- 1601      | 1730           | 1613    | 1654- 1615        | 1655 |
| -C=O stretch (aromatic)  | 1514            | 1546- 1511        | 1509            | 1512           | 1515    | 1559- 1516        | -    |
| C-O-C stretch (ether)    | 1250            | 1247              | -               | 1250           | 1250    | 1250              | 1250 |
| C-O stretching           | 1104            | 1110              | 1121            | 1105           | 1118    | 1108              | 1106 |
| Strange bonds            | -               | -                 | -               | -              | -       | -                 | -    |
| C≡N stretching           | 2360            | 2361              | -               | 2359- 2342     | -       | -                 | -    |
| C-F                      | 1387            | -                 | -               | 1377           | -       | -                 | -    |
| C-Cl                     | 832- 797        | -                 | -               | 795- 768       | -       | -                 | -    |

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[44]

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الموجز

الهدف من هذه البحث إلقاء الضوء على وضع غش المبيدات في السوق المصري عن طريق رصد مبيد الأفات (أبامكتين) كمثال، والذي واستخدامه على نطاق واسع في مصر من حيث الغش والتزيف، من خلال دراسة وتحليل مستحضرات أبامكتين (سبع عينات) تم جمعها من السوق المصرية. تم دراسة المبيدات المزيفة في مصر خلال عدة إستمارات استبيان لكافة العاملين في منظومة المبيدات (المزارع - التاجر - الباحثين في قطاع المبيدات). أظهرت النتائج التي تم الحصول عليها أن بعض العينات تحتوي على المواد الفعالة التي تقلل من النتائج المعتادة المزيفة، بينما كانت النتائج غير مطابقة في الثمانية عينات. أظهرت النتائج عدم التخزين المتناسب (بالتفاوت مع العينات التي تم تخزينها في الثمانية عينات، حيث أظهرت هذه العينات تفاؤلًا ملحوظًا. وشملت هذه العينات 

كلمات المفتاحية: المبيدات المقلدة، المبيدات غير المشروعة وغير القانونية، أبامكتين، مطيات الكتلة، الأنشطة تحت الحماية، الخواص الطبيعية

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