Contents lists available at Science-Gate

International Journal of Advanced and Applied Sciences

Journal homepage: http://www.science-gate.com/IJAAS.html

Strategic preparation of tetraconazole from PTFE waste: Addressing the impending PFAS regulations

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ARTICLE INFO

Article history:
Received 9 March 2025
Received in revised form
21 July 2025
Accepted 16 August 2025

Keywords:
Triazole fungicides
Tetraconazole
PFAS
PTFE waste
Tetrafluoroethylene addition

ABSTRACT

Stricter global regulations on PFAS compounds have created a need for sustainable waste management solutions. In this study, we propose a method to synthesize the fungicide tetraconazole using PTFE waste as a raw material. This approach converts a difficult-to-recycle waste into a valuable product, helping to address environmental challenges. The process involves the controlled pyrolysis of PTFE waste to produce tetrafluoroethylene (TFE), which then reacts with 2-(2,4-dichlorophenyl)-3-(1H-1,2,4-triazol-1-yl)propan-1-ol (compound 4-b) in a benzotrifluoride solution under optimized conditions. This reaction yields tetraconazole with a GC purity of 99.25%. The findings support circular economy practices and align with emerging strict PFAS regulations.

1. Introduction

Triazole fungicides help agricultural growers fight plant diseases, but face challenges with dosing and safety concerns (Corrêa-Junior et al., 2024; Li et al., 2024; Aoujil et al., 2024). Most triazole compounds inhibit crop growth and shouldn't be used during the seedling stage, except for cereals (Shahid et al., 2024; Jeschke, 2024). In general, triazole fungicides can have varying degrees of inhibitory effects on crops and are typically not recommended for use during the seedling stage, with the exception of cereal crops (Shahid et al., 2024; Jeschke, 2024). Surprisingly, tetraconazole stands out as relatively safe because it does not interfere with the normal biosynthesis of gibberellins in plants or inhibit their normal growth (Li et al., 2024; Jiang et al., 2024). This allows it to be used at any stage of crop development, including under high-temperature and high-humidity conditions, without causing phytotoxicity (Degradi et al., 2024). Detailed information regarding its physical and chemical properties can be found in Table 1.

Tetraconazole, a second-generation triazole fungicide, inhibits the biosynthesis of fungal ergosterol, thereby preventing the formation of bacterial cell membranes and ultimately leading to the death of fungal cells (Ahmed et al., 2021; Sieiro-Sampedro et al., 2020). It strongly inhibits fungal hyphal growth, germination, and conidia formation, effectively suppressing the spread and development of diseases (Dutilloy et al., 2022). As a highly efficient, low-toxicity, broad-spectrum fungicide, primarily tetraconazole, it is employed for the prevention and control of diseases affecting cucumbers, strawberries, melons, rice, and ornamental roses and shows remarkable efficacy against powdery mildew and superior activity in combating sheath blight and anthracnose, making it a preferred choice for comprehensive plant protection (Elmer and White, 2018; Moudgal and Sobel, 2003). Furthermore, the outstanding dual solubility of tetraconazole in both water and fat enhances its ability to effectively penetrate a plant's protective waxy layer (Rutkowska et al., 2023). Upon application, tetraconazole is rapidly absorbed and strongly translocated internally, ensuring an even distribution throughout the plant tissues. This characteristic not only enhances preventive measures but also provides exceptional curative efficacy against fungal infections (Tsuda et al., 2004; Bossche et al., 1987).

In summary, tetraconazole, a new broad-spectrum triazole fungicide with internal absorption,

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https://doi.org/10.21833/ijaas.2025.09.014
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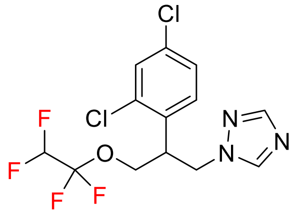
is expected to remain popular in the future market among pesticide researchers, producers, fruit and vegetable farmers, and agricultural sectors, especially welcomed by socialized plant protection and control service organizations (Patrício et al., 2008). The growing number of tetraconazole product registrations, expanded crop applications, broader control targets, enhanced mixed-product usage, and development of eco-friendly formulations underscore its significant value in the future plant protection market. Tetraconazole and its mixtures are promising for growth prospects (Peng et al., 2013; Chew and Than, 2016). Despite its commercial importance, the production of tetraconazole faces significant challenges related to its synthesis. The key to the synthesis lies in addressing the introduction of the tetrafluoroethyl group, as illustrated in Fig. 1.

Table 1: Physical and chemical properties of tetraconazole including structure, formula, appearance, and solubility characteristics

Product name	Tetraconazole
Chemical name	2-(2,4-Dichlorophenyl)-3-(1H-1,2,4-triazol-1-yl)propyl-1,1,2,2-tetrafluoroethylether
CAS No.	112281-77-3
Molecular formula	$C_{13}H_{11}Cl_2F_4N_3O$
Molecular weight	372.15 Dalton
Appearance	Colorless to light yellow viscous liquid
Molecular structure	
Boiling point	438.4 ± 55.0 °C at 760 mmHg
Solubility	0.156 g/L in water (20.0 °C), easy to dissolve in organic solvents

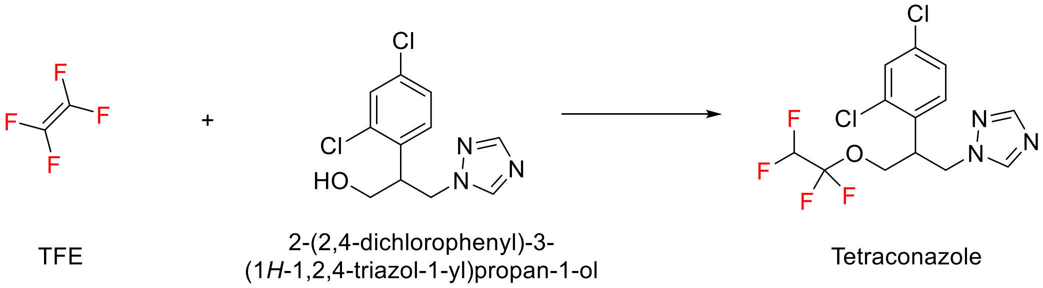


Fig. 1: Reaction scheme showing the addition of TFE to compound 4-b to form tetraconazole

The established synthesis route for tetraconazole, as illustrated in Fig. 1, encounters a significant challenge: The use of tetrafluoroethylene (TFE) (Václavík et al., 2018). TFE is highly hazardous and cannot be transported easily (Tang and Tsui, 2024; Puts et al., 2019). Additionally, factories equipped to produce TFE typically prefer not to engage in the production of fine chemicals, adding another layer of complexity (Rust et al., 2024). Consequently, the critical intermediate, 2-(2,4-dichlorophenyl)-3-(1H-1,2,4-triazol-1-yl)propan-1-ol, must be shipped to these specialized facilities for the essential TFE etherification step. This reliance on outsourcing results in limited autonomy and adaptability for manufacturers of tetraconazole.

Polytetrafluoroethylene (PTFE), widely recognized by the trade name Teflon®, is extensively utilized for its exceptional nonstick characteristics and remarkable chemical resistance (Lee et al., 2024; Silva et al., 2024; Karhan and Arslan, 2024). However, its durability and resistance to degradation make it difficult to recycle, leading to significant environmental challenges (Chai et al., 2024; Qiu et al., 2025). On the other hand, per- and polyfluoroalkyl substances (PFASs) are a class of chemicals that include PTFE and are under scrutiny because of their persistence in the environment and potential health risks (Pu et al., 2025; Aborode et al., 2025; Renella et al., 2025). As regulatory bodies worldwide tighten restrictions on PFAS, alternative methods for utilizing PTFE waste are urgently needed (Gao et al., 2025; Chu et al., 2024; Donley et al., 2024).

Tetraconazole is an antifungal compound with broad-spectrum activity against various fungi that affect crops (De Mello-Sampayo et al., 2024). Its synthesis typically involves complex organic reactions, making its production costly and resource-intensive (Samia et al., 2024). By utilizing PTFE waste as a starting material, this study aims to create a sustainable and cost-effective method for producing tetraconazole.

This study aims to address these dual challenges by developing a novel process that utilizes PTFE waste as a source of TFE for tetraconazole synthesis. The objectives of this study are to (1) establish optimal reaction conditions for the addition of TFE to compound 4-b, (2) demonstrate the feasibility of using pyrolyzed PTFE waste as a TFE source, and (3) evaluate the efficiency and quality of the resulting tetraconazole product. The successful implementation of this approach would represent a significant advancement in both sustainable waste management and agrochemical synthesis.

2. Results and discussion

The solvent and compound 4-b were placed in a high-pressure reactor, as shown in Fig. 2. An alkaline catalyst was added, and freshly bottled TFE was injected into the high-pressure reactor. The reaction was investigated by gas chromatography (GC) under the following conditions:

- Chromatography column: 1000 mm × 3 mm (id) glass column, internally filled with 5% SE-30 Chrom W-AW DMCS filler, with particle sizes ranging from 150 μm to 180 μm;
- Column temperature: 200 °C;
- Vaporizer chamber temperature: 250 °C;
- Detecting chamber temperature: 250 °C;
- Carrier gas flow rate: (N_2) 30 mL/min;
- Hydrogen flow rate: 50 mL/min;
- Air flow rate: 500 mL/min;
- The injection volume was 1.0 μL.

A series of optimization experiments was conducted to determine the ideal conditions for the addition reaction between TFE and compound 4-b. The parameters investigated included the solvent type, alkaline catalyst, reaction temperature, pressure, and 4-b concentration. Initial experiments were performed using bottled TFE to establish baseline conditions before transitioning to PTFE waste as the TFE source.

2.1. Selection of the solvent

We initially selected potassium hydroxide as the base, following protocols from hexaflumuron synthesis (Zhang et al., 2010; Yin et al., 2023). We tested various solvents at 15% concentration of compound 4-b, running reactions at 100 °C and 2.0 MPa for 5 hours. Table 2 shows the GC analysis results.

Fig. 2: High-pressure reactor used for TFE addition reactions with temperature and pressure control systems

Table 2 indicates that benzotrifluoride significantly outperforms other solvents, yielding 27.06% tetraconazole compared with 5.27–15.13% with alternative solvents. This superior performance may be attributed to the unique combination of polarity, boiling point (102 °C), and compatibility with fluorinated compounds of benzotrifluoride. However, the overall yield remains suboptimal, suggesting that the alkaline catalyst (potassium hydroxide) may not provide sufficient activation of the hydroxyl group in compound 4-b for nucleophilic reaction with TFE. Therefore, it is crucial to continue researching and optimizing different bases to improve the reaction efficiency.

Table 2: Comparison of six solvents for the TFE addition reaction showing impurity levels, residual 4-b, and tetraconazole yields by GC analysis

Entry	Solvent name	GC results (%)		
		Total impurity	4-b	Tetraconazole
1	Toluene	2.25	85.63	12.12
2	Nitrobenzene	2.77	91.96	5.27
3	Benzotrifluoride	2.01	70.93	27.06
4	Chlorobenzene	1.98	85.59	12.43
5	Dimethyl benzene	2.63	86.38	10.99
6	1,3-Dichlorobenzene	2.12	82.75	15.13

2.2. Selection of the alkaline catalyst

Benzotrifluoride was used as the solvent, while other variables were maintained to study the impact of various alkaline catalysts on the reaction. The findings are presented in Table 3.

Table 3 unequivocally illustrates that replacing potassium hydroxide with potassium carbonate as the alkaline catalyst significantly increased the yield of tetraconazole. Therefore, potassium carbonate is clearly a more efficacious and appropriate choice for this reaction.

2.3. Selection of the reaction temperature

The effect of temperature on the reaction was investigated using potassium carbonate as the alkaline catalyst and benzotrifluoride as the solvent, with the concentration of compound 4-b maintained at 15%. The reaction was carried out at a pressure of 2.0 MPa. The experimental findings are summarized in Table 4. Table 4 reveals that the most favourable combination of TFE and compound 4-b occurs at a reaction temperature of 100 °C. This observation aligns with the synthesis experience of hexaflumuron, suggesting that increasing the temperature and reducing the pressure within certain boundaries promote the addition of TFE (Ibrahim et al., 2024). Consequently, the initially chosen 100 °C was validated as the optimal temperature for the reaction.

Table 3: Effect of different alkaline catalysts on the TFE addition reaction, showing impurity, residual 4-b, and tetraconazole yield percentages

Entry	Alkaline catalyst name	GC results (%)		
		Total impurity	4-b	Tetraconazole
1	Potassium hydroxide	2.01	70.93	27.06
2	Sodium hydroxide	2.28	75.79	21.93
3	Potassium carbonate	1.97	51.26	46.77
4	Sodium carbonate	2.05	58.62	39.33
5	Sodium bicarbonate	1.73	79.15	19.12
6	Lithium hydroxide	2.56	74.89	22.55

2.4. Selection of reaction pressure

At a fixed reaction temperature of 100 °C, potassium carbonate was employed as the alkaline catalyst, and benzotrifluoride was used as the solvent (with an addition to achieve a 4-b concentration of 15%).

A study was carried out to assess how changes in pressure affect the reaction outcome. The findings from this examination are summarized in Table 5. Table 5 clearly shows that the most favourable addition effect was achieved when the reaction pressure was maintained at 1.0 MPa, implying that an initial experimental pressure of 2.0 MPa was not optimal.

Table 4: Temperature optimization (70-120 °C) data for the TFE addition reaction showing impurity, residual 4-b, and tetraconazole yields

Entry	Reaction temperature (°C)	GC results (%)		
		Total impurity	4-b	Tetraconazole
1	70	1.34	65.83	32.83
2	80	2.02	57.79	40.19
3	90	1.42	57.03	41.55
4	100	1.97	51.26	46.77
5	110	2.29	59.62	38.09
6	120	2.45	64.78	32.77

Table 5: Pressure optimization (0.0-5.0 MPa) data for the TFE addition reaction showing impurity, residual 4-b, and tetraconazole yields

Entry	Reaction pressure (MPa)	GC results (%)		
		Total impurity	4-b	Tetraconazole
1	0.0	2.42	62.83	34.75
2	1.0	1.78	47.00	51.22
3	2.0	1.97	51.26	46.77
4	3.0	2.07	54.64	43.29
5	4.0	1.88	60.44	37.68
6	5.0	2.54	65.00	32.46

2.5. Selection of the 4-b concentration

Compound 4-b, i.e., 2-(2,4-dichlorophenyl)-3-(1H-1,2,4-triazol-1-yl) propan-1-ol, can dissolve up to 25% in benzotrifluoride. The increased concentration of 4-b leads to increased production efficiency, making it crucial to investigate its impact on the reaction.

The effects of different 4-b concentrations on the reaction were studied at a constant temperature of 100 °C and pressure of 1.0 MPa, and the results are presented in Table 6.

Table 6: Effect of compound 4-b concentration (5-25%) on the TFE addition reaction showing impurity, residual 4-b, and tetraconazole yields

Entry	4-b concentration (%)	GC results (%)		
		Total impurity	4-b	Tetraconazole
1	5	1.71	52.96	45.33
2	10	1.65	44.38	53.97
3	15	1.78	47.00	51.22
4	20	2.73	48.76	48.51
5	25	2.19	56.93	40.88

As shown in Table 6, the addition of TFE to compound 4-b yields the best results when the concentration of 4-b is set at 10%. This finding indicates that the initial experimental conditions with a 4-b concentration of 15% were not optimal.

On the basis of the results of the single-factor experiments, freshly bottled TFE was replaced with PTFE waste. Importantly, when PTFE waste is used instead of bottled TFE, the TFE necessary for the reaction is supplied by the pyrolysis of PTFE waste. Therefore, increasing the original reaction temperature is necessary.

2.6. PTFE waste pyrolysis and integration with tetraconazole synthesis

On the basis of the optimized reaction conditions identified in previous experiments (benzotrifluoride solvent, potassium carbonate catalyst, 100 °C reaction temperature, 1.0 MPa pressure, and 10% 4-b concentration), the process was adapted to utilize PTFE waste as the TFE source. The pyrolysis of PTFE waste was conducted at temperatures between 600 °C and 700 °C in the first tubular reactor, generating TFE monomers that were immediately introduced to the reaction mixture in subsequent reactors. This in situ generation of TFE eliminates the hazards associated with TFE storage and transportation.

The integration of PTFE pyrolysis with tetraconazole synthesis requires careful temperature control across the reactor train to ensure efficient TFE generation without degradation of other reaction components. The recycling system allowed for nearly complete conversion of compound 4-b (residual<1.5%) while maximizing TFE utilization from the PTFE waste. The process achieved a total conversion efficiency of approximately 95% relative to the theoretical yield on the basis of the fluorine content of the consumed PTFE waste.

2.7. Yield consistency, purification losses, and product stability

To evaluate process reliability, five consecutive batches were produced using the optimized conditions. The overall yield remained consistent at 94.3±1.2%, demonstrating excellent reproducibility. Purification losses were quantified at each step: Filtration (2.1%), solvent recovery (1.3%), and distillation (2.2%), for a cumulative loss of 5.6%. The final tetraconazole product showed excellent stability, with no detectable degradation (<0.1%) when stored at room temperature (25 °C) for six months.

Accelerated stability testing at 40 °C and 75% relative humidity revealed only 0.8% degradation after three months, suggesting strong product stability. These results project a shelf-life exceeding 24 months when stored appropriately, supporting reliability and durability under recommended storage conditions.

3. Conclusions

This study successfully demonstrates a strategic approach to preparing tetraconazole from PTFE waste, offering multiple benefits that address pressing industrial and environmental challenges. The developed methodology provides several significant advantages.

3.1. Economic viability analysis

A preliminary cost comparison between conventional tetraconazole synthesis and our PTFE waste-based approach reveals significant economic advantages. The conventional route requiring commercially produced TFE has an estimated production cost of approximately $45-50 per kilogram of tetraconazole, with TFE accounting for 40% of this cost. In contrast, our PTFE waste-based synthesis reduces production costs to approximately $32-35 per kilogram, representing a 30% cost reduction. This saving stems primarily from replacing commercial TFE ($28/kg) with PTFE waste ($0.8-1.5/kg), while additional processing costs (pyrolysis, enhanced safety measures) only partially offset these savings. Furthermore, as PFAS regulations become stricter, the cost of PTFE waste disposal is expected to increase, potentially making our approach even more economically favorable as waste generators may provide PTFE at negative costs to avoid disposal fees.

3.2. Industrial scale-up and safety considerations

The transition from laboratory to industrial scale production presents several challenges that require careful consideration. For safe industrial implementation, we recommend: (1) Continuous process monitoring with real-time TFE detection systems to prevent dangerous accumulation; (2) Reactor design with pressure relief systems calibrated specifically for fluorinated compounds; (3) Implementation of zone-based safety protocols with specialized ventilation systems in TFE generation areas; (4) Automated emergency shutdown systems triggered by pressure/temperature anomalies; and (5) Dedicated personnel training for handling fluorinated intermediates.

The process is amenable to industrialization through a modular reactor design that separates the high-temperature pyrolysis zone from the subsequent reaction zones, minimizing thermal stress on the catalyst and reaction components. We estimate that a production facility processing 500 kg/day of PTFE waste could produce approximately 1750 kg/day of tetraconazole, sufficient to meet regional agricultural demands. The pyrolysis and reaction systems should be housed in separate fireproof chambers with independent safety controls, while maintaining process integration through carefully engineered transfer systems.

Future research should focus on optimizing the PTFE waste pyrolysis conditions to maximize the TFE yield and exploring other high-value applications for PTFE-derived fluorochemicals. Additionally, a comprehensive life cycle assessment of this process compared with that of conventional tetraconazole synthesis would further establish its environmental benefits.

3.3. Environmental impact assessment

A preliminary environmental impact analysis of our process compared to conventional tetraconazole production demonstrates significant ecological benefits. Based on established life cycle assessment methodologies for fluorochemical production, we estimate our approach reduces greenhouse gas emissions by approximately 40% (from 12.5 to 7.3 kg CO_2-eq per kg of tetraconazole). This reduction stems primarily from avoiding virgin TFE production, which typically requires energy-intensive electrofluorination. Additionally, our process decreases landfill burden by approximately 3.2 kg of PTFE waste per kg of tetraconazole produced.

Water usage is reduced by an estimated 35% compared to conventional synthesis routes, while the potential for harmful fluorochemical release into the environment is minimized through our closed-loop recycling system, which captures and reuses unreacted TFE.

By integrating waste management with chemical production, our approach embodies a practical example of circular economy principles in agrochemical manufacturing, providing a template for similar waste valorization strategies in the chemical industry.

4. Experimental section

4.1. General methods

All solvents and raw materials employed are commercially available industrial standards and are utilized without any special pretreatment. All the experimental procedures were conducted in ambient air unless otherwise specified. To the best of our knowledge, there is no publicly documented literature regarding the synthesis of tetraconazole utilizing waste derived from PTFE.

4.2. Synthetic route

The synthesis of tetraconazole involves an addition reaction between TFE and 2-(2,4-dichlorophenyl)-3-(1H-1,2,4-triazol-1-yl)propan-1-ol. The chemical reaction equation is depicted in Fig. 3.

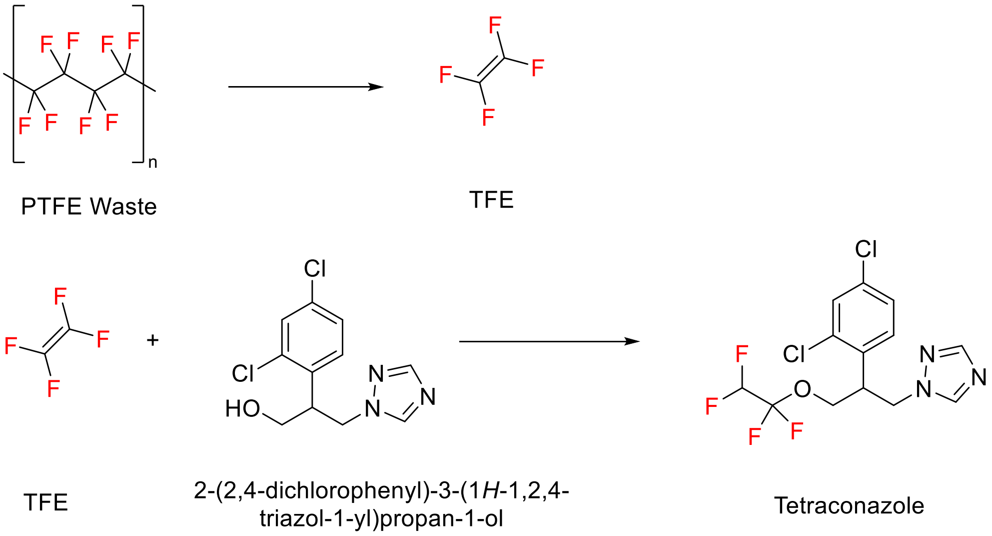


Fig. 3: Two-stage process for tetraconazole synthesis: Pyrolysis of PTFE waste to generate TFE, followed by addition reaction with compound 4-b

4.3. Experimental process

Compound 4-b is a white solid that needs a suitable solvent for the reaction. We conducted single-factor experiments to optimize reaction conditions using bottled TFE. Our results showed benzotrifluoride as the best solvent and potassium carbonate as the optimal catalyst. The ideal conditions were 100 °C, 1.0 MPa pressure, and 10% concentration of compound 4-b.

For the formal experiments, freshly bottled TFE was replaced with PTFE waste. The experimental process utilized a custom-designed reactor system consisting of five serially connected tubular reactors (316 stainless steel, 1.5 m length × 50 mm inner diameter) with independent temperature control systems. PTFE waste powder (particle size<0.5 mm), potassium carbonate (1.2 eq. relative to compound 4-b), and compound 4-b (10% w/v in benzotrifluoride) were evenly blended in a 5 L jacket mixing vessel equipped with a high-shear impeller operating at 500 rpm. The mixture is subsequently pumped via a rotor pump into two serially connected tubular reactors. Next, the reaction stream is conveyed through three consecutive tubular reactors to yield the preparation liquid, which is temporarily stored in a finished product receiving tank. If compound 4-b is not fully consumed, the preparation liquid should be recycled back into the reaction system until the residual amount of 4-b falls below 1.5%. The preparation mixture was removed, centrifuged, and the cake was filtered to isolate unreacted PTFE waste and potassium carbonate. The separated materials were washed with methanol, and they were dried before recycling. The filtrate was composed of tetraconazole, trace amounts of unreacted 4-b, potassium carbonate, and benzotrifluoride. The filtrate was concentrated to recycle the benzotrifluoride solvent and then distilled under reduced pressure to obtain pure tetraconazole.

The total yield of tetraconazole was calculated via the following equation:

$$\text{Total Yield} = \frac{m_3}{(m_2 - m_1) \times \frac{372.14}{100.02}} \times 100\%$$

where, m_3: weight of the finished product of tetraconazole; m_2: The feed weight of PTFE waste; m_1: The residual weight of PTFE waste.

Fig. 4 illustrates that the final product of tetraconazole is obtained as a colourless viscous liquid, exhibiting a high GC purity of 99.25%.

Fig. 4: Final tetraconazole product (99.25% GC purity) synthesized from PTFE waste, showing characteristic appearance

List of abbreviations

Chrom W-	Chromosorb W acid-washed
AW DMCS	dimethylchlorosilane (chromatography filler)
CO_2-eq	Carbon dioxide equivalent
DMI	Demethylase inhibitor
GC	Gas chromatography
id	Inner diameter
L	Liter
MPa	Megapascal
N_2	Nitrogen
OSL	Optically stimulated luminescence
PFAS	Per- and polyfluoroalkyl substances
PTFE	Polytetrafluoroethylene
rpm	Revolutions per minute
SE-30	Silicone elastomer 30 (stationary phase for chromatography)
TFE	Tetrafluoroethylene
UV-C	Ultraviolet C
w/v	Weight per volume
µL	Microliter
µm	Micrometer

Acknowledgment

The authors acknowledge funding from Liaoning Longtian Chemical Technology Co., Ltd. and thank Dr. Puivun Chai for his contributions to this research.

Compliance with ethical standards

Conflict of interest

The author(s) declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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