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"Optimized Reaction System for Efficient Diethyl Phosphite Synthesis"

Mohd. Shamim Choudhary[1*], Dr. Ravi W. Tapre[2]
[1]Student, University of Mumbai, Thane, Maharashtra 400608, India*
[2]Assistant Professor, Department of Chemical Engineering, Datta Meghe College of Engineering, Mumbai University, Airoli, Navi Mumbai, Maharashtra, 400708, India

***Abstract**: The invention concerns an improved method for the technical production of diethyl phosphite and ethyl chloride, wherein the starting material ethanol is mixed with method-specific additives and the mixture is subsequently reacted with phosphorus trichloride. The coupled product ethyl chloride is thereafter isolated and utilized based on its achieved purity. The present invention provides an improved process for the production of diethyl phosphite using ethanol as a solvent and phosphorus trichloride as the principal reactant. The process performance was assessed across varying ethanol molar ratios and temperature profiles, which were shown to influence product yield and by-product formation. A key feature of the invention is the controlled removal of hydrogen chloride generated during the reaction, thereby suppressing the formation of undesirable phosphite by-products. Through the application of an inert-gas purge and optimized reaction parameters, the process enables the efficient formation of diethyl phosphite with enhanced purity and yield. The invention thus offers a more reliable and industrially advantageous approach to the large-scale production of diethyl phosphite.*

***Keywords**: diethyl phosphite, ethyl chloride, phosphorus trichloride, ethanol, hydrogen chloride etc.*

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I. Introduction:

The production of diethyl phosphite $(EtO)_2P(O)H$ by reacting phosphorus trichloride (PCl_3) with ethanol (EtOH or C_2H_5OH) is a two-stage process that typically involves the formation of an intermediate, which is then converted into the final product.

The overall reaction is generally represented as:

$$PCl_3 + 3C_2H_5OH \longrightarrow (C_2H_5OH)_2P(O)H + C_2H_3Cl + 2HCl$$

This method is often part of a larger industrial process and yields diethyl phosphite and ethyl chloride as a co-product, along with hydrogen chloride (HCl).

1. Reaction Pathway

The reaction to form diethyl phosphite does not happen directly in a single step but is believed to proceed through the intermediate formation of triethyl phosphite ($(EtO)_3P$)

Step 1: Formation of Triethyl Phosphite (Esterification)

When PCl_3 is reacted with ethanol in the presence of a base (like a tertiary amine, R_3N e.g., triethylamine or dimethylaniline) which acts as an HCl scavenger, the main product is triethyl phosphite (a P (III) compound).

Step 2: Conversion to Diethyl Phosphite (Partial Alcoholysis/Hydrolysis)

In the absence of a base, or if the intermediate triethyl phosphite is allowed to react with the co-product hydrogen chloride (HCl) or trace amounts of water (H_2O), it undergoes conversion to the more stable P(V) species, diethyl phosphite.

The most common conversion routes are:

Reaction with HCl**:** The HCl produced in the overall process can react with the triethyl phosphite to displace one ethyl group, yielding diethyl phosphite and ethyl chloride.

$$(C_2H_5O)_3P + HCl \longrightarrow (C_2H_5O)_2P(O)H + C_2H_5Cl$$

Reaction with Water (Hydrolysis): Industrially, diethyl phosphite is also often produced by the partial hydrolysis of triethyl phosphite

$$(C_2H_5O)_3P + H_2O \longrightarrow (C_2H_5O)_2P(O)H + C_2H_5OH$$

The resulting diethyl phosphite, $(EtO)_2P(O)H$, exists predominantly in the P(V) phosphonate form (shown) rather than the P(III) phosphite form, exhibiting tautomerism.

2. Experimental Considerations

- Stoichiometry**:** The overall net reaction consumes **3** moles of ethanol for 1 mole of PCl_3
- Conditions**:** The reaction is highly sensitive to conditions. To Favor the formation of the desired diethyl phosphite, the reaction is typically run under controlled temperatures and in a way that allows the HCl or a small, controlled amount of water to be present or introduced to drive the second step.
- Byproducts: The key challenge is separating the volatile byproducts (HCl and C2H5Cl) from the liquid product, which is usually done by distillation. The co-production of C_2H_5Cl is a defining feature of this industrial synthesis route.

Properties:

Chemical Name	diethyl phosphonite
Molecular Formula	$(C_2H_5OH)_2P(O)H$
Molecular Weight	138.10
Melting point	-70°C
Boiling point	50-51 °C
Density	1.07 g/mL at 25 °C
solubility	Soluble in Water
color	Colorless to Water-white/Clear

II. Literature Review:

Common Synthesis Routes: The established commercial route for producing diethyl phosphite involves the reaction of phosphorus trichloride (PCl_3) with ethanol (EtOH)

$$PCl_3 + 3C_2H_5OH \longrightarrow (C_2H_5OH)_2P(O)H + C_2H_3Cl + 2HCl$$

A related but distinct reaction utilizes a base (e.g., triethylamine) to scavenge the HCl byproduct, leading primarily to the formation of triethyl phosphite ($(C_2H_5O)_3P$), which can then be converted to DEP via reaction with HCl or partial hydrolysis. Alternative industrial processes involve reacting triethyl phosphite with phosphorous acid, which can be optimized to produce high-purity, low-acidity DEP.

Hydrophosphonylation (Addition Reactions): This is the most crucial application, involving the addition of DEP across unsaturated bonds to form C-P bonds

- Pudovik Reaction (Abramov Reaction): Addition to aldehydes or ketones, typically base-catalyzed, to synthesize alpha-hydroxyphosphonates. These products are important intermediates for biologically active compounds.
- Kabachnik-Fields Reaction (or Three-Component Reaction): A one-pot condensation involving an aldehyde/ketone, an amine, and DEP to synthesize alpha-aminophosphonates. This reaction is highly efficient (atom-economical) and is used in the synthesis of peptide mimetics and biologically relevant molecules

C-P Bond Formation:

- Michaelis-Becker Reaction**:** DEP is deprotonated with a strong base (like potassium *tert*-butoxide) to form a reactive phosphonate anion, which then undergoes nucleophilic substitution with an alkyl halide (R-X) to form a P-C bond (an alkylphosphonate)
- Hirao Reaction**:** This is a palladium-catalyzed cross-coupling of DEP (or its anion) with aryl or vinyl halides (Ar-X or R-CH=CH-X) to form arylphosphonates and vinylphosphonates. This method is vital for synthesizing medicinally relevant phosphonates.
- Transesterification: DEP can react with higher-boiling alcohols to exchange the ethyl groups, providing access to a variety of dialkyl phosphites

- Nucleoside Synthesis**:** DEP is a key starting material for synthesizing important antiviral drugs, such as acyclic nucleoside phosphonates (ANPs) like Adefovir and Tenofovir, which are used to treat HIV and Hepatitis B.

Advanced Process Design: The present invention relates to an improved process for the production of diethyl phosphite utilizing ethanol as a solvent and phosphorus trichloride as a key reactant. The process has been evaluated under various conditions, including differing molar ratios of ethanol, which were found to influence both the product yield and the formation of by-products. Variations in temperature profiles were likewise studied to determine their effect on reaction completion.

A critical aspect of the process is the continuous removal of hydrogen chloride generated during the reaction, as the presence of hydrogen chloride promotes the undesired formation of triethyl phosphite. In the improved method, the reaction mixture is subjected to a controlled inert-gas purge to facilitate the steady displacement of hydrogen chloride and volatile by-products, thereby favouring formation of the desired compound.

Under optimized conditions employing an ethanol-to-phosphorus trichloride molar excess and a controlled low-temperature regime, the target diethyl phosphite is obtained with high purity and yield. The process thus provides a reliable and industrially advantageous method for producing diethyl phosphite with reduced by-product formation and improved operational efficiency. Experimental data given bellow which collected in lab.

Sr. No	Ethanol Mole	Temperature	Diethyl Phosphite Yield	Triethyl Phosphite Yield	Purging	Remark
1	1	25-30 ^{O}C	35%	65%	Not Provided	Exotherm Observed
2	1.5	25-30 ^{O}C	25%	75%	Not Provided	More side product form
3	1	10-15 ^{O}C	30%	60%	Not Provided	More side product form
4	1.5	10-15 ^{O}C	25%	75%	Not Provided	More side product form
5	1	10-15 ^{O}C	75%	-	N_2	Unreacted
6	2	0-5 ^{O}C	85%	-	N_2	Unreacted
7	3	0-5 ^{O}C	99%	-	N_2	-

In the course of the reaction, the evolving hydrogen chloride and chloroethane are directed into a separate collection vessel containing chilled water, thereby enabling their controlled absorption or capture. The experimental setup may include a reaction flask equipped with a thermowell, mechanical agitation, a cooling system, and a gas-transfer assembly incorporating an outlet conduit and an inert-gas purging device. The process additionally provides an opportunity for the recovery of chloroethane as a valuable by-product suitable for commercial utilization. As chloroethane is a flammable gas, appropriate precautions are observed to ensure safe handling and to maintain the material at a safe distance from ignition sources.

III. Conclusion:

The experimental evaluation demonstrates that the improved process significantly enhances the efficiency and selectivity of diethyl phosphite production when appropriate molar ratios of ethanol and phosphorus trichloride are employed in conjunction with controlled low-temperature conditions and continuous removal of hydrogen chloride. The data confirm that inadequate hydrogen chloride removal and higher reaction temperatures lead to increased formation of triethyl phosphite and other side products, whereas the application of an inert-gas purge at reduced temperatures markedly improves the purity and yield of the desired product. Optimal results were obtained at higher ethanol molar excess under low-temperature operation, yielding diethyl phosphite of high purity with minimal by-product formation. The process further enables the controlled capture of hydrogen chloride and chloroethane, the latter of which may be recovered as a commercially valuable co-product. Accordingly, the invention provides a robust and industrially advantageous method for the efficient production of diethyl phosphite while simultaneously facilitating safe and effective management of gaseous by-products.

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The authors declare that they have no Conflict of Interest.

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Cover Letter

This manuscript is the authors' original work and has not been published nor has it been submitted simultaneously elsewhere. All authors have checked the manuscript and have agreed to the submission.

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The paper has been submitted with full responsibility, following due ethical procedure, and there is no duplicate publication, fraud, plagiarism. None of the authors of this paper has a financial or personal relationship with other people or organizations that could inappropriately influence or bias the content of the paper. This article does not contain any studies with human participants or animals performed by any of the authors.

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