Cost-Effective Teacher

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A Simple and Efficient Ozone Generator

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Ozonolysis in the educational laboratory has been primarily a tool used in structure determination (*1–3*). Instrumental methods of analysis have effectively eliminated the need for these oxidation reactions and removed ozonolysis from the classroom. However, oxidation is still an important topic in introductory courses and ozonolysis can play an important role in describing this concept. Typically, however, the preparative uses of ozone imply use of a commercial apparatus. Commercial ozone generators are often unwieldy in size and inefficient in their production of ozone. On the other hand, most of the ozone generators reported in this *Journal*, with a single exception (*4*), are for small-scale production of derivatives for structural determination and are unsuitable for the safe generation of the quantities of ozone needed for an oxidation reaction (*6–13*). We report herein the construction of an inexpensive, efficient, and compact ozonolysis apparatus that is a convenient alternative to the more expensive generators currently on the market.

Ozone is produced in a silent electrical discharge within which electrons with sufficient kinetic energy split the O=O bond upon impact (*14*). In general, when an alternating current voltage is applied to a sample of oxygen, ozone production doubles as the frequency of the current is doubled. The amount of ozone generated varies exponentially with the applied voltage. The half-life of ozone is more than 5000 s at temperatures less than 100 °C and tends to increase with decreasing ozone concentration. Mudd and co-workers (*15*) were able to generate ozone by passing O_2 at a flow rate of 0.33 $cm^3\ s^{-1}$ (20 $cm^3\ min^{-1}$) through a silent electric discharge. In their experiment, ozone was produced at a rate of approximately 2 μmol min^{-1}.

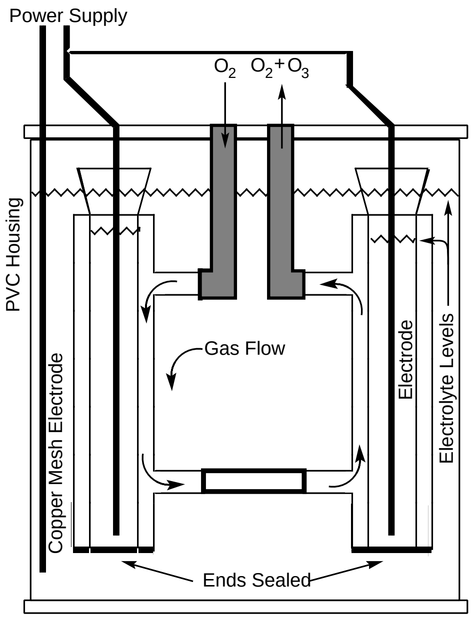


Figure 1. Schematic diagram of the ozone generation cell.

The Apparatus

A schematic design of an ozone generator suitable for lab scale investigations using a corona discharge has been reported in the literature (*5, 16*). The device we developed is simpler in construction and readily assembled from components commonly found in any laboratory. It is based on the published schematic but, rather than being custom glassblown, is constructed of standard West condensers. Two condensers, each with one ground glass joint removed and the end sealed, are connected in series with Tygon tubing and suspended in a custom-made PVC container. This container is constructed from 11.5-cm o.d., 10-cm i.d. pipe. It is sealed with an O-ring to a bottom flange and has a removable press-fit top (Fig. 1). The length of each condenser is approximately 20 cm. The innermost cavity of each condenser, which is normally used to condense solvent vapor, is filled with electrolyte. One electrode of the discharge, a 0.25-inch copper tube, is inserted into the solution in this inner cavity and subsequently connected to the power source. The other electrode of the discharge, which is constructed from copper mesh, is inserted into the electrolyte solution surrounding the entire condenser assembly. The copper mesh may be electrically grounded or connected to the power source as necessary and, typically, a 0.3 M $CaCl_2$ solution is used as the electrolyte. When generating ozone at temperatures below 20 °C, a stainless steel dewar is substituted for the PVC container and electrolyte solutions of 4.5 M $MgCl_2$ or 6.5 M $CaCl_2$ are used owing to the lower freezing points of these solutions. The power supply, shown in Figure 2, consists of a step-up transformer for the line voltage and a set of contact points operated by a second transformer. The power supply output is limited to less than 0.5 A and, on average, is approximately 20 kV at an unregulated frequency near 60 Hz.

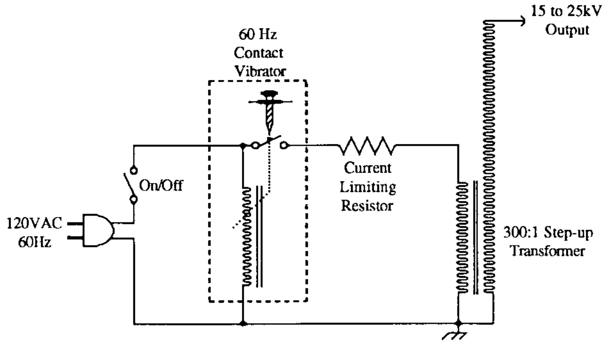


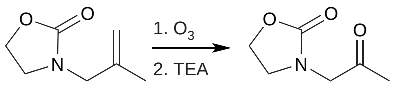
Figure 2. Power supply schematic diagram.

Ozone is produced by passing zero grade oxygen, 99.8% purity, through the cooling jacket of the condenser. The maximum concentration of ozone obtained with the feed oxygen and discharge near room temperature was approximately 4% v/v at a flow rate of 0.33 mL s^{-1}. A marginal increase in ozone concentration was measured when the discharge temperature was cooled to −20 °C. Decreasing the feed gas temperature further by passing it through a pentane/liquid nitrogen slurry bath (approximately −150 °C) at a flow rate of up to approximately 25 mL s^{-1} resulted in concentrations up to 10% v/v of ozone in oxygen.

Photometric detection at 254 nm allowed the determination of ozone concentration in the effluent stream. (The O_3 absorption coefficient at 254 nm is 134 ± 2 atm^{-1} cm^{-1} at 273 K and 123 ± 2 atm^{-1} cm^{-1} at 298 K [*17–24*].) Alternatively, a titrimetric technique that involves bubbling the effluent gas through an aqueous solution of potassium iodide may also be used to determine ozone concentration (*25*).

Advantages of this ozone generating system are that it does not require expensive, high-purity oxygen and that it can be constructed from common glassware. The power source involves only three electrical components and the specifications of those components are not critical to the operation of the device.[1] The simplicity of design and efficiency of ozone production offered by this apparatus should make it ideal for use in advanced laboratory settings. The preparation of 3-(2-oxopropyl)-1,3-oxazolidin-2-one (2) is illustrative of the use of the apparatus described above. Many other olefinic substrates could be oxidized in like fashion. In this case, the starting material is obtained by N-alkylation of the sodium salt of 2-oxazolidinone. The procedure features the use of triethylamine for ozonide decomposition, offering the benefit of easy workup and purification of the desired carbonyl product.

General Procedure for Ozonolysis



1 **2**

A 50-mL pear-shaped flask was charged with 0.342 g (1.55 mmol) of methallyl oxazolidinone (1) and 35 mL of freshly distilled (CaH_2) CH_2Cl_2. The solution was cooled to −78 °C and O_3 was bubbled through it. To effect addition of ozone, the exit port of the ozone generator was connected with Tygon tubing to a piece of glass tubing that terminated in a glass frit. Ozone was delivered to the reaction solution using this fritted tube, the fritted end being submerged in the solvent. After approximately 30 min the solution was blue, indicating the presence of excess ozone. The ozone delivery tube was removed from the reaction and the flask was placed under a slightly positive pressure of N_2 on an inert atmosphere line. Triethylamine (TEA) was added (0.41 mL, 3.09 mmol) and the solution was gradually warmed to room temperature and stirred overnight (*26*). The reaction was washed with brine and dried ($MgSO_4$), and the solvent was removed in vacuo.

*3-(2-Oxopropyl)-1,3-oxazolidin-2-one (**2**)*

Chromatography of the crude product (EtOAc) yielded the desired compound quantitatively. ^1H NMR ($CDCl_3$, 300 MHz) δ 4.40 (dd, 2H, *J* = 8.06 Hz), 4.11 (s, 2H), 3.66 (dd, 2H, *J* = 8.06 Hz), 2.18 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 202.3, 158.8, 62.1, 53.1, 44.9, 26.8; TLC R_f 0.19 (75% EtOAc/hexanes).

Safety Warnings

CAUTION: Ozone is a highly reactive and toxic gas. It rapidly, and sometimes violently, reacts with organic molecules. Chronic exposure and exposure to high concentrations may cause respiratory difficulties and eye irritation. The apparatus described in this report should only be operated with proper ventilation, preferably in a fume hood. Generation of ozone requires a high-voltage discharge. Care should be taken to physically and electrically isolate the high-voltage components of this apparatus from any nearby solvents.

Note

1. The following electrical components are suitable for power supply construction: EG&G, Inc. Step-up transformer (Model TR-153), Allied Electronics 120V Repeat Cycle Timer (Model 519-4005) and 10 kΩ current limiting resistor. However, any similar components will be acceptable.

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