

計畫名稱：工業園區廢水及地下水污染調查分析研究

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中文摘要

本計畫主要目標為建立四種標的化合物在廢水及地下水中之檢測方法，此四種化合物為四氫呋喃、異丙醇、二甲基甲醯胺，以及二甲亞風。本研究團隊透過國內外相關文獻的回顧、過去的實務經驗，以及實際的試驗分析結果，得到檢測此四種化合物的方法，分別為分析四氫呋喃及異丙醇的共沸蒸餾/氣相層析儀火燄離子偵測器法，與檢測異丙醇、四氫呋喃、二甲基甲醯胺及二甲亞風的固相微萃取/氣相層析質譜儀法。

以共沸蒸餾/氣相層析儀火燄離子偵測器法測試去離子水樣品，1公升樣品經共沸蒸餾約60分鐘，收取前10毫升之蒸餾液，將1微升蒸餾液注入氣相層析儀，以膜厚1 μ m、內徑0.53mm、30米長之DB-WAX層析管柱分析。實驗結果顯示，異丙醇和四氫呋喃均可達80%以上之前處理效率；而異丙醇檢量線濃度在24 μ g/L與393 μ g/L間線性關係可達0.995，四氫呋喃檢量線濃度在27 μ g/L與443 μ g/L間線性關係亦可達0.993。本研究依照環檢所公告之方法進行方法偵測極限測試與確認，四氫呋喃之偵測極限為2.72 μ g/L，而異丙醇為1.59 μ g/L。固相微萃取/氣相層析質譜儀法使用Carboxen/PDMS纖維，以30 $^{\circ}$ C進行20分鐘之浸入式吸附，並於250 $^{\circ}$ C之氣相層析儀注射埠脫附4分鐘後，樣品以60米長、膜厚1.8 μ m、內徑0.32mm之DB-624層析管柱分析。實驗結果顯示，異丙醇、四氫呋喃、二甲基甲醯胺及二甲亞風感應因子的相對百分偏差都小於20%，而線性範圍異丙醇為200 μ g/L至1500 μ g/L、四氫呋喃為20 μ g/L至150 μ g/L、二甲基甲醯胺為0.5mg/L至5mg/L、二甲亞風為30mg/L至450mg/L。研究結果也依照單一實驗室方法驗證程序完成精密度、準確度、樣品保存期限等測試。

本研究的另一個工作為調查科學工業園區廠商，在製程中使用危害性或仍未明之化學藥品與溶劑之種類與使用量。由於這些資料的取得甚為困難，因此本研究除了以問卷調查方式來了解園區廠商化學品使用的狀況外，亦透過主管機關(環保署、地方環保局)擁有的清理計畫書與申報資料來彌補問卷回收率低的不足。根據三種方式對新竹科學園區部份廠商調查的結果，發現除了酸類、丙酮、異丙醇等純溶劑以外，多種成分組成之光阻劑、去光阻劑、顯影劑也是大量使用的化學品。

本研究最後一項工作乃在於真實樣品的分析，分析樣品的基質包含地下水、廢水與製程廢液。經吹氣捕捉法的分析，地下水樣品檢測出之污染物以含氮揮發性有機化合物為主；污水處理廠之進流水及放流水則含有丙酮、二甲基硫、甲苯，其中某一調勻池樣品更檢測出二甲基甲醯胺。因其為本研究建立檢測方法之化合物，正好使用此樣品進行添加測試，以確認所建立方法之適用性。另三個製程廢液則分別檢測出含有異丙醇及二甲亞風。相同地，這些樣品也被使用進行添加及重覆添加測試，以確認所建立品保規範之適用性。

總結本計畫之執行，包含了建立以共沸蒸餾/氣相層析儀火燄離子偵測器法檢測異丙醇、四氫呋喃，與固相微萃取/氣相層析質譜儀法檢測異丙醇、四氫呋喃、二甲基甲醯胺及二甲亞風，並逐一經

過驗證程序建立了品保品管規範，也透過化學品調查，選取了含有待測化合物的真實樣品，經測試驗證了方法的適用性，並完成了研擬二篇檢測方法草案。期望本研究對於環境保護領域地下水、廢水中水溶性有機污染物之檢測能有所貢獻與幫助。

The main purpose of this study is to establish the testing methods for Isopropanol, Tetrahydrofuran, Dimethylformamide, and Dimethyl sulfoxide in wastewater and groundwater. Based on the previous experiences and extensive literature review, the research team developed two methods to test these four compounds. One is using Azeotropic Distillation Procedure and Gas Chromatographic/Flame Ionization to determine Isopropanol and Tetrahydrofuran, and another is using Solid Phase Micro Extraction and Gas Chromatographic/Mass Spectrometric (GC/MS) to determine Isopropanol, Tetrahydrofuran, Dimethylformamide, and Dimethyl sulfoxide.

The procedure for azeotropic distillation is as follows. After spiking with analytes, the reagent water sample was gone through the azeotropic distillation for 60 minutes, and then 10mL distillate was collected in which 1mL distillate was injected into gas chromatograph. A 30-meter of DB-WAX column with 0.53mm inner diameter and 1mm film was used for analysis. Pretreatment efficiency for both isopropanol and tetrahydrofuran can be reached up to 80%. Linearity was 0.995 for isopropanol and 0.993 for tetrahydrofuran. Detection limits of this method were 1.59 mg/L and 2.72 mg/L for isopropanol and tetrahydrofuran, respectively. For Solid Phase Micro Extraction, 75mm of Carboxen/PDMS fiber was used. After being immersed in 8mL sample for adsorption at 30°C for 20 minutes then the fiber was placed at 250°C GC injection port for desorption 4 minutes. A 60-meter DB-624 column with 0.32mm inner diameter and 1.8 um film was used for analysis. The relative standard deviations in linearity of four compounds were below 20%. Detection limits of this method were 32.8 ug/L, 2.3 ug/L, 37.0 ug/L, and 2.8 mg/L for isopropanol, tetrahydrofuran, dimethyl formamide, and dimethyl sulfoxide, respectively. Accuracy, precision, and sample holding time were also tested according to the designated verification procedure.

Another task in this study was to conduct a survey to understand the amount of some suspicious hazardous chemicals and solvents, which are commonly used in the processes of companies within the Science Based Industrial Park. In addition to the questionnaire, the cleanup declaration files of manufactures, which submitted to the local authorities were also collected and analyzed for the completeness of the survey.

Real sample analysis was also included in this study for verifying the methods developed. The samples includes both groundwater and wastewater samples, which comes from the industrial park. The results showed that chlorinated VOCs were detected in ground water by purge and trap/GCMS. Acetone, dimethyl sulfide and toluene were the main pollutants in the influent and effluent of the wastewater treatment plant. Dimethyl formamide was detected in one of the influent samples and isopropanol and dimethyl sulfoxide were detected in another two samples. These samples were used for spike analysis in order to verify the applicability of the testing methods developed in this study.

In summary, this study has successfully developed methods for analyzing four compounds, namely Isopropanol, Tetrahydrofuran, Dimethylformamide, and Dimethyl sulfoxide. By implementing the verification procedure, the quality control and quality assurance criterion were also established. Real samples contain target compound in different matrix were also analyzed to verify the applicability of the methods. And the drafts of the standard procedures for the testing methods developed in this study were also proposed. Hopefully this study can enhance the understanding and capability of analyzing dissolved organic pollutants in the groundwater and wastewater samples.