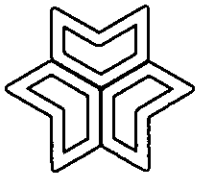

Environmental Engineering

Technical Report

Trace Organics in Composted Sewage Sludge
Part 2. Survey
July, 1994

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Submitted to the

Commonwealth of Massachusetts
Department of Environmental Protection
Bureau of Resource Protection
Thomas Powers, Acting Commissioner
Andrew Gottlieb, Director
Office of Watershed Management

ABSTRACT

The objective of this research was to produce a list of the trace organic compounds prevalent in sewage sludge compost and to attempt to determine the fate and removal mechanisms of trace organics during sludge composting. This was accomplished in three steps. First, a detailed literature review was completed examining information and collected data on the levels of trace organic compounds in municipal wastewater sludge and sludge compost. The fates of these compounds during composting was also investigated. The second step involved a survey of wastewater treatment plants currently practicing composting in western Massachusetts. From the list of treatment plants generated during the survey, three facilities were chosen for preliminary sampling. Finished compost from the three facilities (Holyoke, Springfield, and Williamstown) was collected and analyzed for trace organic contamination. The information gathered during the preliminary sampling permitted a rational choice of study site for the third part of this project. The third step involved collecting and analyzing raw and finished compost at the chosen plant (Holyoke). Five samples were collected over a nine-month period. The results of the first step are presented as separate volume (Part 1), while the results of steps two and three are presented in this technical report.

The information obtained during the literature review and the results from both the preliminary and final sampling all suggest that very few trace organic compounds are present in composted sewage sludge. In addition, research has shown that many of the trace organics belonging to the list of EPA priority pollutants are either biodegraded to some extent or volatilized during composting. Therefore, any trace organics which may be present in raw compost will probably be degraded and reduced to very low concentrations. The main conclusion reached from these findings and the results of this

project is that composted sewage sludge does not contain trace organic contamination which could otherwise limit its application to land or use as a beneficial sludge product.

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1.0 INTRODUCTION

1.1 Objectives

In response to the proposed 503 standards, this study, funded by the Massachusetts Division of Water Pollution Control (MDWPC), was undertaken to produce a list of the trace organics prevalent in sewage sludge and to attempt to determine the fate and removal mechanisms of trace organics during composting. This was accomplished in three steps. First, a detailed literature review was completed examining information and collected data on the levels of trace organic contaminants in municipal wastewater sludge and sludge compost. The specific topics addressed were:

1) What are the sources of trace organics in wastewater?
2) What is the fate of trace organics in wastewater treatment plants and how are they incorporated into the sludge?
3) Which trace organics are typically found in sewage sludge?
4) What is the fate of trace organics during composting?
5) Which trace organics (if any) are typically found in finished compost?

The results of this first step are reported in Part 1 of this this technical report (7).

The second step was to survey wastewater treatment plants currently practicing composting in western Massachusetts. The objective was to gather any available data on trace organic contamination in sludge and compost. This information permitted a rational choice of study site for the third part of this project. Some preliminary sampling at the best perspective plants was necessary during this phase. The third step involved collecting and analyzing parent sludge and final compost at the chosen plant. Five samples were collected over a nine-month period. The results of steps two are three are presented in this technical report.

During the course of this project, the final Part 503 rule was published on February 9,1993 in the Federal Register (58FR9248). After obtaining the results from the National Sewage Sludge Survey (5) and conducting extensive scientific peer review, EPA realized that levels of trace organics in sewage sludge from wastewater treatment plants had significantly decreased since the 1978 Fate Study (upon which the proposed 503 pollutant limits were based) (1,2). Therefore, numerical limits for organic compounds were completely omitted from the final Part 503 rule. However, since organic pollutants may be included in the list of pollutants that EPA will identify for regulation in second stage (Round 2) rulemaking, this study focused on the organic compounds originally listed in the proposed 503 regulations under sections 503.13 (agricultural land) and 503.22 (distribution and marketing). These compounds, referred to in this report as the trace organic compounds of concern, are presented in Table 1-1.

Table 1-1 TRACE ORGANIC COMPOUNDS OF CONCERN

Aldrin/dieldrin (total)
Benzo (a) pyrene
Chlordane
DDT/DDE/DDD (total)
Dimethyl Nitrosamine
Heptachlor
Hexachlorobenzene
Hexachlorobutadiene
Lindane
Polychlorinated Biphenyls (PCBs)
Toxaphene
Trichloroethylene

The overall objective of this study was to provide some fundamental information on the fate of trace organics during sludge composting. The data should be valuable to

monitoring and regulating treatment and disposal of sludge and compost. The background literature is presented in Part 1 (7) while this report presents the results of a data collection and sampling program for composting facilities in western Massachusetts.

2.0 SAMPLE COLLECTION AND ANALYSIS

2.1 Preliminary Sampling

A survey of wastewater treatment plants which were practicing composting in western Massachusetts was performed to identify possible candidates for preliminary sampling. The facilities eventually chosen for preliminary sampling were the Hoosac Water Quality District wastewater treatment plant in Williamstown, the Holyoke composting plant, and the Springfield wastewater treatment plant. These three facilities were chosen because they were facilities which were representative of three different community sizes. Williamstown is located in a rural community, Holyoke is a medium-sized city, and Springfield is a larger city. Also, different methods of composting are utilized at each of the three facilities. The facility in Williamstown uses the aerated static pile method, while the other two facilities are of the in-vessel type. The Holyoke facility uses a system designed by International Process Systems (IPS). The IPS system is an enclosed, agitated, multi-bay composting process. The Springfield facility uses a cylindrical tower, plug-flow, in-vessel composting reactor. All three facilities primarily use wood chips as the bulking agent.

Finished compost was collected at the Holyoke, Springfield, and Williamstown facilities in early October, 1992. At the Holyoke facility, a composite sample was collected from the end of bays 4 and 5. At the Springfield facility, a composite sample was collected from a 30-day cured pile, and at the Williamstown facility the composite sample was collected from compost which had been cured for approximately 6 to 7 weeks.

2.2 Raw and Finished Compost Sampling

After the initial sampling at the facilities, it was decided that the Holyoke facility was the best plant for further sampling. The main reason that the Holyoke facility was

chosen was that the agitated, multi-bay configuration allows for easy sampling at different points during the composting process. Thus, a single batch of sludge can be continuously monitored and sampled throughout the composting cycle in this system. Through such an analysis, the measurement of trace organic removal during composting is possible. However, for reasons discussed later, this sort of analysis was not performed at the Holyoke facility.

During an eight month period, four more samples were collected at the Holyoke facility (November, 1992; January, 1993; March, 1993; and June, 1993). In each sampling, composite samples were collected at the back (raw compost) and the front (finished compost) of a composting bay.

2.3 Sample Collection

For each sample collected, a composite sample was obtained. This was accomplished by digging approximately 1 foot into the compost at several points in the compost pile and collecting grab samples with a large stainless steel spoon (except at the Williamstown facility where a backhoe dug several feet into the curing pile). The grab samples (approximately five large "scoops" each) were then mixed in a stainless steel pail with the spoon. The mixed grab samples were then put into glass jars and vials using a stainless steel spatula. The glass jars and vials were then placed into a cooler containing ice packs and quality control samples. After collection, the composite samples were immediately transported to Tighe & Bond Environmental Laboratory in Westfield, Massachusetts for analysis.

Prior to each sampling, the stainless steel pail, spoon, and spatula, and cooler were obtained from Tighe & Bond. On each occasion, the stainless steel instruments were previously decontaminated by Tighe & Bond and placed into plastic bags. The bags remained sealed until just prior to actual sample collection. When two samples (raw and finished compost) were collected at the Holyoke facility, the stainless steel instruments

were decontaminated between samplings with methanol and rinsed with de-ionized water. Raw samples were always collected after the finished compost to prevent any possible contamination of the finished compost samples with trace organics present in the raw compost samples.

2.4 Analytical Methods

Sludge and compost samples were analyzed for volatile organics and extractable organics by Tighe & Bond Environmental Laboratory in Westfield, Massachusetts. Samples were analyzed by EPA Methods 8240 (volatile organics), 8270 (semivolatile organics), and 8080 (pesticides and PCBs). Compounds which are detected by these EPA methods are listed in Appendix A. Tighe & Bond also tested the samples for total Kjeldahl nitrogen, ammonia nitrogen, nitrite/nitrate nitrogen, percent solids, dimethyl disulfide, methyl sulfide, dimethyl trisulfide, and trimethylamine. These later compounds are of interest because they are significant odor-causing contaminants.

Method 8240 is used to determine volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, and can be used to quantify most volatile organic compounds that have boiling points below 200° C and that are insoluble or slightly soluble in water. The volatile organics are extracted from the solid matrix using the purge-and-trap procedure (Method 5030) and are analyzed by gas chromatography/mass spectrometry (GC/MS).

Method 8270 is used to determine the concentration of semivolatile organic compounds in extracts prepared from all types of solid waste matrices. This method can be used to quantify most neutral, acidic, and basic organic compounds that are soluble in methylene chloride. The compounds are extracted from solids by the sonication process (Method 3550), which ensures intimate contact of the sample matrix with the extraction solvent, and analyzed by GC/MS.

Method 8080 is used to determine the concentration of various organochlorine pesticides and polychlorinated biphenyls (PCBs). In this method, the compounds are also extracted from the solid matrices by the sonication process and are analyzed by gas chromatography. All referenced methods are from reference (4).

3.0 RESULTS AND DISCUSSION

Summaries of the results from the preliminary and final sampling are shown in Tables 3-1 and 3-2. It is evident from the results that very few trace organic compounds were detected in either the parent sludge (raw compost) or finished compost samples.

Table 3-1 PRELIMINARY SAMPLING RESULTS

Sample location and date	Compounds detected in finished compost	Concentration (µg/kg) dry wt.
1) Holyoke, 10/1/92	Dimethyl disulfide	50
2) Springfield, 10/1/92	Lindane	1100
	Bis (2-ethylhexyl) phthalate	*51,000
	Benzoic acid	58,000
	Acetone	*2,800
	Methylethyl ketone (MEK)	2,600
	Dimethyl disulfide	1,800
3) Williamstown, 10/8/92	Bis (2-ethylhexyl) phthalate	*860
	4-Methylphenol	730
	Methylethyl ketone (MEK)	260
	Acetone	*1500
	Toluene	15

*-Also detected in laboratory blank at a similar level

Table 3-2 FINAL SAMPLING RESULTS

Sample location and date	Raw/Final compost	Compounds detected in compost	Concentration (µg/kg) dry wt.
4) Holyoke 11/19/92	Raw	Bis (2-ethylhexyl) phthalate	11,000
		Acetone	82,000
		Methylethyl ketone (MEK)	79,000
		Toluene	340
		Total xylenes	150
		Dimethyl disulfide	640
	Final	Bis (2-ethylhexyl) phthalate	37,000
		Pyrene	3,200
5) Holyoke 1/7/93	Raw	Bis (2-ethylhexyl) phthalate	*18,000
		Acetone	73,000
		Methylethyl ketone (MEK)	220,000
		Chloroform	84
		Ethylbenzene	31
		Toluene	420
		Total xylenes	180
	Final	Bis (2-ethylhexyl) phthalate	*39,000
		Dimethyl disulfide	720
6) Holyoke 3/18/93	Raw	Bis (2-ethylhexyl) phthalate	51,000
	Final	Bis (2-ethylhexyl) phthalate	59,000
		Dimethyl disulfide	360
7) Holyoke 6/23/93	Raw	Bis (2-ethylhexyl) phthalate	36,000
		Acetone	19,000
		Methylethyl ketone (MEK)	15,000
	Final	Bis (2-ethylhexyl) phthalate	46,000

*-Also detected in laboratory blank at a similar level

Samples were also tested for nitrite/nitrate nitrogen, ammonia nitrogen, percent solids, and total Kjeldahl nitrogen. The results from these analyses are shown in Tables 3-3 and 3-4. These results suggest that nitrite/nitrate nitrogen and total Kjeldahl nitrogen decrease during composting, while ammonia nitrogen and percent solids increase during the process.

Table 3-3 NITROGEN CONTENT AND PERCENT SOLIDS (PRELIMINARY SAMPLING)

Sample location and date	Nitrite/nitrate Nitrogen (mg/kg) dry wt.	Ammonia Nitrogen (mg/kg) dry wt.	Percent solids	Total Kjeldahl Nitrogen (mg/kg) dry wt.
Holyoke, 10/1/92	<10	4,800	67.4	21,000
Springfield, 10/1/92	<20	4,800	73.1	41,000
Williamstown, 10/8/92	<10	1,300	50.4	8,600

Table 3-4 NITROGEN CONTENT AND PERCENT SOLIDS (FINAL SAMPLING)

Sample location and date	Raw/Final compost	Nitrite/nitrate Nitrogen (mg/kg) dry wt.	Ammonia Nitrogen (mg/kg) dry wt.	Percent solids	Total Kjeldahl Nitrogen (mg/kg) dry wt.
4) Holyoke 11/19/92	Raw	NA	NA	33.9	NA
	Final	NA	NA	68.5	NA
5) Holyoke 1/7/93	Raw	<30	3,200	32.9	24,000
	Final	<10	4,300	65.6	18,000
6) Holyoke 3/18/93	Raw	<40	2,400	31.2	23,000
	Final	<20	5,500	56.0	20,000
7) Holyoke 6/23/93	Raw	<30	3,700	44.7	15,000
	Final	29	5,000	72.7	12,000

The most commonly detected compound in both the raw and finished compost samples was Bis (2-ethylhexyl) phthalate. Phthalic acid esters are one of the most widely used classes of chemicals today, mainly as plasticizers but also in cosmetics, inks, insect repellents, pesticides, etc. In the National Sewage Sludge Survey (NSSS), Bis (2-ethylhexyl) phthalate was detected nationally in 62 percent of the sludge samples at a

mean concentration of approximately 75,000 µg/kg. In another study EPA study (3), Bis (2-ethylhexyl) phthalate had the highest concentration of any of the semi-volatile compounds in the sludge analyzed. In this study, concentrations of Bis (2-ethylhexyl) phthalate ranged from 11,000 to 51,000 µg/kg in the raw compost samples collected at the Holyoke facility. Considering the fact that raw compost consists of sludge mixed with a bulking agent (typically at a ratio of 2-3 volumes of bulking agent to one volume of sludge but at a 1:1 ratio at Holyoke), the concentrations found in this study are consistent with the national average.

Of the chlorinated pesticides and PCBs, the only compound detected was lindane (detected only once at the Springfield facility). However, it is possible that these pollutants may have been present in the samples at extremely low concentrations. Table 3-5 summarizes some of the results from the NSSS (5) The table shows the compounds detected and the range of concentrations the compounds were measured at. The last column shows the detection limits at which Tighe & Bond was able to measure these organic compounds at in the raw compost in this study. This table illustrates the fact that even if the trace organic compounds of concern were present in the Holyoke raw compost at the concentrations measured in the NSSS, most of them would not have been detected in this study. The concentrations measured in the NSSS were so low that very few laboratories are able to detect such trace quantities. Since lindane was detected only once and at a very high concentration (much higher than the concentrations measured in the NSSS), it is possible that the pesticide may have been present in the bulking agent and not in the sludge. Pesticides are commonly used by homeowners and can end up in yard waste (from which wood chips are made). Yard waste may also contain other trace organic contaminants. Harrad et al. (6) found significant concentrations of hexachlorobenzene in composted yard waste.

Other organic compounds detected were methylethyl ketone (MEK), acetone, toluene, pyrene, chloroform, ethylbenzene, total xylenes, benzoic acid, and 4-methyl

phenol. Dimethyl disulfide, a compound associated with odors at composting facilities, was also detected several times.

Originally, the intent of this study was to monitor and sample batches of sludge throughout the composting process at the Holyoke facility so that the reductions in trace organics could be calculated during a composting cycle. This information would have aided in determining the possible fates and removal mechanisms for the individual trace organic compounds of concern. However, the scarcity of trace organics found in the raw compost samples collected prevented such an analysis from being conducted.

Table 3-5 NSSS RESULTS AND TIGHE & BOND DETECTION LIMITS

Compound	Percent Detected	Actual times detected	Range of values (µg/kg)	Tighe & Bond detection limits (µg/kg)
Aldrin	4	8/198	18-45	300
Benzo (a) Pyrene	3	6/200	671-24703	5,000
Chlordane	1	1/198	489	1500
DDD	1	1/198	391	300
DDE	2	4/198	30-190	300
DDT	4	7/198	15-121	300
Dieldrin	3	5/198	12-47	300
Heptachlor	1	1/198	23	300
Hexachlorobenzene	0	0/200	-	5000
Hexachlorobutadiene	0	0/200	-	5000
Lindane	1	2/198	72-76	300
PCB-1016	0	0/198	-	3000
PCB-1221	0	0/198	-	3000
PCB-1232	0	0/198	-	3000
PCB-1242	0	0/198	-	3000
PCB-1248	11	21/198	43-5203	3000
PCB-1254	5	10/198	312-9347	3000
PCB-1260	10	19/198	31-4006	3000
Toxaphene	0	0/198	-	6000
Trichloroethylene	4	7/200	24-3302	50

It should be pointed out that a direct comparison between the raw and final compost samples collected at the Holyoke facility for this study cannot be made. In other words, percent reductions for trace organic compounds cannot be calculated from this data. Since the raw and finished samples were taken on the same day, it is impossible to know what compounds (and at what concentrations) were present in the finished compost when the composting cycle for this particular slug of compost began (approximately 21 days prior to sampling). It cannot be assumed that the concentrations of the trace organics measured in the raw compost on the day of sampling are representative of the concentrations that were present when the composting cycle for the finished compost began. Also, the reduction of mass of the composting material has an effect on the concentration of trace organics because the concentrations of trace organics are usually reported as μg/kg. In other words, there is a certain mass of pollutant per mass of composting material. Since a significant amount of organic material is converted to carbon dioxide and water, the resultant concentrations of trace organics can thus be altered (even if the amount of the compound remains the same). For example, if there is 1 microgram of an organic pollutant in a kilogram of composting mass, the concentration of the organic is 1.0 μg/kg. However, if 50 percent of the composting mass is converted to carbon dioxide and water and the amount of pollutant remains the same, the concentration of the pollutant is now 2.0 μg/kg (1.0 μg/0.50 kg = 2.0 μg/kg). Therefore, it is possible that the concentrations of recalcitrant trace organics in compost may increase during the composting process. Assuming that the concentration of Bis (2-ethylhexyl) phthalate is relatively constant in the compost reactor feed, this may explain why the concentration of Bis (2-ethylhexyl) phthalate is higher in the final compost than in the raw compost (see Table 3-2).

During preliminary sampling at the Williamstown facility, the operator (George Heisler) kindly provided results from sampling of finished compost that was conducted in April and August of 1992. The analyses (very similar to the analyses performed by Tighe & Bond in this study) were performed by Alpha Analytical Laboratories in Westborough, Massachusetts. The results of the sampling were also very similar to the results obtained in this study. The only trace organic compound which was detected was Bis (2-ethyl hexyl) phthalate. Pesticides, PCBs, and numerous other organic compounds were tested for but were not found.

As was discussed in Part 1 of this technical report (literature review), many compounds of environmental concern are amenable to biodegradation in a composting process. It might be possible to further remove trace organic compounds by optimizing the potential of the composting process for a given trace contaminant. Measure such as addition of a primary carbon source, nutrients, and/or bioaugmentation with a special culture of microorganisms could promote additional biodegradation of a given contaminant.

4.0 CONCLUSIONS

Based on the results of Part 2 of this study, the following may be concluded:

1) Of the sludges sampled in this study, few trace organic compounds were found in either raw sludge or finished compost. Compounds which were detected were usually present at extremely low concentrations. This is in agreement with the literature survey reported in Part 1 of this report.

2) The concentrations of those trace organic compounds found in the raw sludge, decreased during composting. However, the compounds were not always completely degraded and a residual may remain in the compost. This is also in agreement with the literature survey reported in Part 1 of this report.

3) It is unclear which removal mechanisms account for the losses of individual trace organics during sewage sludge composting.

4) The limited sampling preformed in this study support the conclusion (of Part 1) that trace organic compounds are not widespread in composted sewage sludge and should not prevent the land application of composted sewage sludge.

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APPENDIX

Compounds Detected by EPA Methods 8240, 8270, and 8080

EPA Method 8240S (volatile organics-solids)

Acetone
Acrolein
Acrylonitrile
Benzene
Bromodichloromethane
Bromoform
Bromomethane
2-Butanone {MEK}
Carbon disulfide
Carbon tetrachloride
Chlorobenzene
Chloroethane
2-Chloroethyl vinyl ether
Chloroform
Chloromethane
Dibromochloromethane
Dibromomethane
1,4-Dichloro-2-butene
Dichlorodifluoromethane
1,1-Dichloroethane
1,2-Dichloroethane
1,1-Dichloroethene
trans-1,2-Dichloroethene
1,2-Dichloropropane
Total 1,3-Dichloropropene
Ethanol
Ethylbenzene
Ethyl methacrylate
2-Hexanone
Iodomethane
Methylene chloride
4-Methyl-2-pentanone
Styrene
1,1,2,2-Tetrachloroethane
Tetrachloroethene
Toluene
1,1,1-Trichloroethane
1,1,2-Trichloroethane
Trichloroethylene
Trichlorofluoromethane
1,2,3-Trichloropropane
Vinyl acetate
Vinyl chloride
Total Xylenes

EPA Method 8270S (semivolatile organics-solids)

Acenaphthene
Acenaphthylene
Acetophenone
Aniline
Anthracene
4-Aminobiphenyl
Benzidine
Benzo (a) anthracene
Benzo (b) fluoranthene
Benzo (k) fluoranthene
Benzo (ghi) perylene
Benzo (a) pyrene
Bis (2-chloroethoxy) methane
Bis (2-chloroethyl) ether
Bis (2-chloroisopropyl) ether
Bis (2-ethylhexyl) phthalate
4-Bromophenyl phenyl ether
4-Chloroaniline
1-Chloronaphthalene
2-Chloronaphthalene
4-Chlorophenyl phenyl ether
Chrysene
Dibenz (a,j) acridine
Dibenz (a,h) anthracene
Dibenzofuran
Di-n-butylphthalate
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
3,3'-Dichlorobenzidine
Diethyl phthalate
p-Dimethylaminoazobenzene
7,12-Dimethylbenz (a) anthracene
Dimethylphenethylamine
Dimethyl phthalate
2,4-Dinitrotoluene
2,6-Dinitrotoluene
Diphenylamine
1,2-Diphenylhydrazine
Di-n-octylphthalate
Fluoranthrene
Fluorene
Hexachlorobenzene
Hexachlorobutadiene
Hexachlorocyclopentadiene
Hexachloroethane
Ideno (1,2,3-cd) pyrene
Isophorone
3-Methylcholanthrene
2-Methylnaphthalene
Naphthalene
1-Naphthylamine
2-Naphthylamine
2-Nitroaniline
3-Nitroaniline
4-Nitroaniline
Nitrobenzene
N-Nitroso-di-n-butylamine
N-Nitrosodimethylamine
N-Nitrosodiphenylamine
N-Nitrosodipropylamine
N-Nitrosopiperidine
Pentachlorobenzene
Pentachloronitrobenzene
Phenanthrene
2-Picoline
Pyrene
1,2,4,5-Tetrachlorobenzene
1,2,4-Trichlorobenzene
Benzoic acid
Benzyl alcohol
4-Chloro-3-methylphenol
2-Chlorophenol
2,4-Dichlorophenol
2,6-Dichlorophenol
2,4-Dimethylphenol
4,6-Dinitro-2-methylphenol
2,4-Dinitrophenol
2-Methylphenol
3-Methylphenol
4-Methylphenol
2-Nitrophenol
4-Nitrophenol
Pentachlorophenol
Phenol
2,3,4,6-Tetrachlorophenol

2,4,5-Trichlorophenol

2,4,6-Trichlorophenol

EPA Method 8080S (organochlorine pesticides and PCBs-solids)

Aldrin
alpha-BHC
beta-BHC
gamma-BHC (lindane)
delta-BHC
Chlordane
4,4'-DDD
4,4'-DDE
4,4'-DDT
Dieldrin
Endosulfan I
Endosulfan II
Endosulfan sulfate
Endrin
Endrin aldehyde
Heptachlor
Heptachlor epoxide
Methoxychlor
Toxaphene
PCB-1016
PCB-1221
PCB-1232
PCB-1242
PCB-1248
PCB-1254
PCB-1269