**STABILITY OF EXTRACTABLE ORGANIC CONSTITUENTS
IN STORED SAMPLES OF GASIFICATION CONDENSATES
INFERRED FROM COMPARISONS OF
GC/MS TOTAL ION CHROMATOGRAMS**

by

L. A. Raphaelian and W. Harrison

MASTER

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ABSTRACT

Samples of water and recycle oil were collected from the prequench tower of the gasifier crude-gas quench system of the HYGAS pilot plant. The samples were stored at refrigerator temperature (2-4°C) for 60 days and at incubator temperature (37°C) for 7 days. A dried oil sample and neutral, acid, base, and composite extracts were prepared at days 0, 29, and 60 for the refrigerated samples and at day 7 for the incubated sample.

The extracts and dried oil were analyzed by capillary column gas chromatography/mass spectrometry on a 50-m OV-101 column. Minor differences in comparative total ion chromatogram traces can be attributed to variations in the concentrations of extraneous plasticizers or to slight differences in column resolution or sample extraction. No significant changes were apparent in comparative chromatogram traces for the refrigerated and incubated sample extracts and the dried oil. Within the limitations of this study, no degradation of organic components was found in the samples refrigerated for 60 days or in the sample stored at incubator temperature for 7 days.

Argonne National Laboratory procedures for check-in, coding, and transfer of samples are also described.

1 INTRODUCTION

1.1 OBJECTIVE

The objective of this study was to determine the changes in organic constituents, extractable by typical acid/base liquid-liquid extraction methods, in bulk samples of coal gasification condensates stored at refrigerator (2-4°C) and incubator (37°C) temperatures. Total ion chromatogram traces were compared to detect any changes.

1.2 BACKGROUND

Although an assessment of sample stability would seem to be mandatory in any study of coal gasification samples, little is known about the

stability of such samples since measuring the stability of a complex mixture is complicated, difficult, and tedious, at best. Furthermore, because there are matrix effects, a measurement of stability would be required for each sample. Normally, one is more interested in product composition than in the origin of the components; it often does not seem important to know if a few of the components in the mixture are the result of decomposition of a specific component or reaction between two or more components in the original mixture. This is "standard practice" in gas chromatography/mass spectrometry (GC/MS) identifications of organics in environmental and process stream samples.

In discussions with a variety of investigators (see acknowledgments) it was found that general practice is to store gasification samples at 2-4°C, where there is "insignificant" degradation of organic constituents. To further inhibit degradation, sodium azide or mercuric chloride is sometimes added. (However, it should be noted that such additives can react or catalyze reactions with certain organics and would interfere with mutagenicity measurements.) Freezing of samples might inhibit degradation better than refrigeration. However, work-up of frozen samples is difficult because ice crystals form and the oil is segregated and tends to adhere to the walls of the vessel.

1.3 GENERAL SCHEME FOR EVALUATION

In this study, fresh samples were stored concurrently at 2-4°C (refrigerator temperature) for 60 days and at 37°C (incubator temperature typically used in bioassays) for 7 days. A sample dried over magnesium sulfate was prepared as well as neutral, acid, and base extracts prepared by liquid-liquid extraction. A composite extract was prepared from the neutral, acid, and base extracts. The foregoing were prepared at days 0, 29, and 60 for the refrigerated samples and at day 7 for the incubated sample. These extracts and the dried oil sample were analyzed by capillary column GC/MS on a 50-m OV-101 column. The total ion chromatograms of each of the 7-day (incubated), 29-day (refrigerated), and 60-day (refrigerated) extracts and dried oil samples were compared to the corresponding total ion chromatograms of the 0-day extracts and dried oil samples. Where differences were apparent, the cause of the differences was investigated by examining the mass spectrum of the extraneous peak.

In such an approach, only those compounds that are extractable and that pass through the 50-m capillary column and transfer line into the mass spectrometer are amenable to analysis. Highly polar organics and certain other compounds that are either not extractable or sufficiently volatile -- such as morpholine, gallic acid, and polynuclear aromatics (PNAs) with 5 or more rings -- will not show up as a peak in the total ion chromatogram. For a more exhaustive study that would include such compounds, better methods for isolation and derivatization would have to be developed. However, the present approach does include the products of oxidative degradation due to air (oxygen) or to biochemical oxidation of such compounds as unsaturated hydrocarbons, lower PNAs, thiophenes, alcohols, and phenols.

1.4 PREVIOUS WORK

The authors have been unable to find any published work on the stability of organic constituents during the storage of bulk samples of coal gasification condensates or similar samples.

2 EXPERIMENTAL PROCEDURES

2.1 SAMPLING

A technician at the HYGAS pilot plant obtained samples of quench water and recycle oil from the prequench tower on the gasifier crude-gas quench system. The samples were taken on May 25, 1978, during plant run 72. Samples were collected in widemouth Boston brown glass bottles that had been rinsed with methylene chloride; the bottles' screw caps had been lined with Teflon.

The water sample, taken at 11:30 a.m., consisted of eight 1-gal bottles filled sequentially and labeled "F Code 1060, 400 Section Quench Water Auto, 406-61." Two 1/2-gal bottles of recycle oil were collected at 11:55 a.m.; each bottle was labeled "Recycled Oil, Location 4.06-42." The water and oil samples were delivered to Argonne within three hours of sampling time.

Average, steady-state plant operating conditions at HYGAS during run 72 were as follows: slurry-drying bed temperature, 607°F; low-temperature reactor bed, 1125°F; high-temperature reactor bed, 1430°F; steam-oxygen gasifier bed, 1680°F; run pressure, 527 psig; and char conversion, 79%.

2.2 SAMPLE CHECK-IN, STORAGE, AND WORK-UP FOR GC/MS ANALYSIS

2.2.1 Check-In and Storage

The water and oil samples were checked in and coded according to the protocols described in the Appendix. Bulk sample identification numbers were assigned as follows:

72/H/G/W/2	Prequench-tower water
72/H/G/O/1	Prequench-tower recycle oil

Samples were stored and worked-up according to the scheme shown in Fig. 1.

2.2.2 Sample Work-up

Quench Water. The following extraction procedure was used for the quench-water sample.

PREQUENCH-TOWER WATER SAMPLE
(72/H/G/W/2)

PREQUENCH-TOWER RECYCLE-OIL SAMPLE
(72/H/G/O/1)

Fig. 1. Scheme for Storage and Work-Up of HYGAS Run 72 Gasifier-Condensate Samples for Sample Stability Analysis (ACL = Analytical Chemistry Laboratory)

- A. Measure and record volume (approximately 3200 mL)
- B. Adjust pH to 2 with HCl
- C. Extract with methylene chloride (250, 100, 100 mL)
- D. Separate layers
 1. Aqueous layer
 - a. Adjust pH to 11 with NaOH
 - b. Extract with methylene chloride (250, 100, 100 mL)
 - c. Dry combined CH_2Cl_2 layer with MgSO_4
 - d. Filter
 - e. Split - 50% to "base fraction"
50% to "combined fractions"
 2. Methylene chloride layer
 - a. Extract with 5% NaOH (100, 100 mL)
 - b. Separate layers
 - (1) Methylene chloride layer
 - (a) Dry with MgSO_4
 - (b) Filter
 - (c) Split - 50% to "neutral fraction"
50% to "combined fractions"
 - (2) Aqueous layer
 - (a) Adjust pH to 2 with HCl
 - (b) Extract with methylene chloride (100, 50, 50 mL)
 - (c) Dry CH_2Cl_2 with MgSO_4
 - (d) Filter
 - (e) Split - 50% to "acid fraction"
50% to "combined fractions"
- E. Concentrate each of the combined, neutral, acid, and base fractions to 25-50 mL in a Kuderna-Danish concentrator.
- F. Bring each fraction to 50.0 mL with methylene chloride.

Recycle Oil. The following procedure was used for work-up of the recycle-oil sample.

- A. Weigh out a 2.0 g aliquot
- B. Add 100 mL 1N HCl
- C. Extract with methylene chloride (100, 50, 50 mL)
- D. Separate layers
 1. Aqueous layer
 - a. Adjust pH to 11 with NaOH
 - b. Extract with methylene chloride (50, 50, 50 mL)
 - c. Dry combined CH_2Cl_2 layer with MgSO_4
 - d. Filter
 - e. Split - 50% to "base fraction"
50% to "combined fractions"

2. Methylene chloride layer

- a. Extract with 1N NaOH (100, 100 mL)
- b. Separate layers
 - (1) Methylene chloride layer
 - (a) Dry with MgSO_4
 - (b) Filter
 - (c) Split - 50% to "neutral fraction"
50% to "combined fractions"
 - (2) Aqueous layer
 - (a) Adjust pH to 2 with HCl
 - (b) Extract with methylene chloride (100, 50, 50 mL)
 - (c) Dry CH_2Cl_2 with MgSO_4
 - (d) Filter
 - (e) Split - 50% to "acid fraction"
50% to "combined fractions"
- E. Concentrate each of the combined, neutral, acid, and base fractions to 25-50 mL in a Kuderna-Danish concentrator
- F. Bring each fraction to 50.0 mL with methylene chloride
- G. Prepare dried oil by dissolving 1.00 g of the sample in methylene chloride, drying with magnesium sulfate, filtering to remove the magnesium sulfate, and making up the solution to 50 mL with methylene chloride

Fractions Provided by Argonne's Analytical Chemistry Laboratory (ACL).

The following fractions were provided by the Argonne Chemical Engineering Division's ACL for GC/MS analysis:

Prequenched-Tower Water

Sample of 25 V 78 (unstored sample) *0-day sample*

- 72/H/G/W/2/ACL/5 - neutral fraction
- 72/H/G/W/2/ACL/3 - acid fraction
- 72/H/G/W/2/ACL/4 - base fraction
- 72/H/G/W/2/ACL/2 - composite
- 72/H/G/W/2/ACL/6 - composite laboratory blank

Sample of 1 VI 78 (stored at 37°C) *7-day sample (incubated)*

- 72/H/G/W/2/ACL/10 - neutral fraction
- 72/H/G/W/2/ACL/8 - acid fraction
- 72/H/G/W/2/ACL/9 - base fraction
- 72/H/G/W/2/ACL/7 - composite

Sample of 23 VI 78 (stored at 2-4°C) *29-day sample (refrigerated)*

- 72/H/G/W/2/ACL/3N - neutral fraction
- 72/H/G/W/2/ACL/3A - acid fraction
- 72/H/G/W/2/ACL/3B - base fraction
- 72/H/G/W/2/ACL/3C - composite

Sample of 24 VII 78 (stored at 2-4°C) 60-day sample (refrigerated)

- 72/H/G/W/2/ACL/4N - neutral fraction
- 72/H/G/W/2/ACL/4A - acid fraction
- 72/H/G/W/2/ACL/4B - base fraction
- 72/H/G/W/2/ACL/4C - composite fraction

Prequench-Tower Recycle Oil

Sample of 25 V 78 (unstored sample) 0-day sample

- 72/H/G/O/1/ACL/2 - dried oil
- 72/H/G/O/1/ACL/5 - neutral fraction
- 72/H/G/O/1/ACL/3 - acid fraction
- 72/H/G/O/1/ACL/4 - base fraction
- 72/H/G/O/1/ACL/6 - composite fraction
- 72/H/G/O/1/ACL/7 - composite laboratory blank

Sample of 1 VI 78 (stored at 37°C) 7-day sample (incubated)

- 72/H/G/O/1/ACL/8 - dried oil
- 72/H/G/O/1/ACL/11 - neutral fraction
- 72/H/G/O/1/ACL/9 - acid fraction
- 72/H/G/O/1/ACL/10 - base fraction
- 72/H/G/O/1/ACL/12 - composite

Sample of 23 VI 78 (stored at 2-4°C) 29-day sample (refrigerated)

- 72/H/G/O/1/ACL/OIL - dried oil
- 72/H/G/O/1/ACL/3N - neutral fraction
- 72/H/G/O/1/ACL/3A - acid fraction
- 72/H/G/O/1/ACL/3B - base fraction
- 72/H/G/O/1/ACL/3C - composite

Sample of 24 VII 78 (stored at 2-4°C) 60-day sample (refrigerated)

- 72/H/G/O/1/ACL/OIL - dried oil
- 72/H/G/O/1/ACL/4N - neutral fraction
- 72/H/G/O/1/ACL/4A - acid fraction
- 72/H/G/O/1/ACL/4B - base fraction
- 72/H/G/O/1/ACL/4C - composite fraction

2.3 ANALYSIS OF ORGANIC CONSTITUENTS

Capillary column GC/MS runs were conducted with a 50-m, OV-101 column on the following samples for the 0-day, 7-day (37°C incubated), 29-day (refrigerated), and 60-day (refrigerated) sample extracts:

Prequench-tower water

1. Neutral
2. Acid
3. Base
4. Composite of above
5. Blank

Prequench-tower recycle oil

1. Neutral
2. Acid
3. Base
4. Composite of above
5. Dried oil
6. Blank

A total of 57 capillary column GC/MS runs on these fractions were made using a Hewlett-Packard 5982A GC/MS equipped with a Hewlett-Packard 5934A data system. The data system consists of a 21MX computer with 32K, 16-bit-word core memory, 7900A dual disc drive with 2.5M bytes/disc memory, 5948A data subsystem (A/D and D/A converters), and Tektronix 4012 display terminal. (A 5830 Hewlett-Packard GC was used in place of the 5700 series GC normally delivered with the 5982A GC/MS.)

Due to the anticipated complexity of the samples to be analyzed, wall-coated open tubular (WCOT) capillary columns were used to afford the greatest possible resolution, even though support-coated open tubular (SCOT) type columns are simpler to use. The WCOT columns are also less susceptible to tailing due to adsorptive effects and also can pass components with higher boiling points such as PNAs, which were of interest in this study. With WCOT columns, leak-proof and low-dead-volume connections are required to avoid tailing of peaks; care was taken to ensure that this was the case. A modified split/splitless Hewlett-Packard Grob-type injection system was used for the capillary columns. (In this study, splitless injection was used.) The end of the column was connected to glass-lined stainless steel tubing with an inside diameter of 0.5 mm. The glass-lined stainless steel tubing was connected directly to a line leading to the mass spectrometer source. Since no separator, such as a jet or membrane separator, was used, all components of a mixture exiting the column entered the mass spectrometer. Therefore, there could not be discrimination in the amount of each component reaching the mass spectrometer, as there can be with separators. Assuming the individual components are not lost in the injection system, the column, or the glass-lined stainless steel tubing transfer line, the amount of each component in the mixture reaching the source of the mass spectrometer is a true representation of the quantity injected on column.

3 RESULTS AND INTERPRETATIONS

3.1 PREQUENCH-TOWER WATER

Figures 2-5 show the total ion chromatograms for the neutral, acid, base, and composite extracts of the fresh, and the 7-, 29-, and 60-day stored samples of prequench-tower water. Little overall difference can be seen in the chromatograms for successive extracts of the same type. Examples of the kinds of differences that were found are given below.

Water/Neutral Fraction (Fig. 2). Peak "A" is apparently not present in the 60-day fraction, and has moved to become a shoulder in the adjacent large peak. Peak number 1 is broad in the 0- and 60-day chromatograms, appears as two peaks with tailing in 7-day sample, and is barely resolved as two peaks with tailing in the 29-day sample.

In the 7-day sample, it can be seen that there are two compounds plus tailing due to phenol, which was not separated from the neutral fraction. In the 29-day sample, resolution is not as good as the 7-day sample since there is more phenol. In the 0-day sample, the phenol is predominant. Other differences in this region are due to alkylated phenols which were not fully separated into the acid fractions.

Peak numbers 2, 3, 4, 5, 6 in the 0- and 29-day samples are plasticizers, which are typical contaminants found in GC/MS analysis.

Although comparisons of the 60-day sample with the 0-, 7- and 29-day samples were difficult due to the lower column resolution, no significant changes could be noted.

Water/Acid Fraction (Fig. 3). The differences in the 0-, 7-, 29-, and 60-day fractions in region 1 are due to differences in resolution, caused possibly by the preceding peak that tails. Peak 2 in the 29-day sample is a phthalate.

Water/Base Fraction (Fig. 4). The differences in region 1 of the 0-, 7-, 29-, and 60-day samples are due to differences in the extraction efficiencies of trace amounts of compounds, in this case alkylated pyridines.

Fig. 2. Total Ion Chromatograms of the Neutral Fraction of 0-Day, 7-Day (37°C Incubated), and 29- and 60-Day (Refrigerator-Stored) Prequench-Tower Water Sample

Fig. 3. Total Ion Chromatograms of the Acid Fraction of 0-Day, 7-Day (37°C Incubated), and 29- and 60-Day (Refrigerator-Stored) Prequench-Tower Water Sample

Fig. 4. Total Ion Chromatograms of the Base Fraction of 0-Day, 7-Day (37°C Incubated), and 29- and 60-Day (Refrigerator-Stored) Prequench-Tower Water Sample

Fig. 5. Total Ion Chromatograms of the Composite of Extracts Fraction of 0-Day, 7-Day (37°C Incubated), and 29- and 60-Day (Refrigerator-Stored) Prequench-Tower Water Sample

The doublet at 2 in the 0-day sample is plasticizer. Peaks 3 and 4 in the 29-day sample are examples of noise spikes.

3.2 PREQUENCH-TOWER RECYCLE OIL

Figures 6-10 show the total ion chromatograms for the neutral, acid, base, composite, and dried-oil extracts of the fresh, and the 7-, 29-, and 60-day stored samples of recycle oil. As was the case with the extracts of the water sample, few changes can be seen. Following are examples of the kinds of differences that were found

Oil/Neutral Fraction (Fig. 6). The inverse peak in region 1 of all fractions is due to the mass spectrometer being turned off while toluene, the major component of the mixture, elutes. Peaks 2, 3, 4, 5 and 6, present only in the 29-day run, are plasticizers.

Oil/Acid Fraction (Fig. 7). The differences in the cluster of peaks are due to differences in extraction and work-up and in column resolution. Peak 1 of the 60-day sample is due to plasticizers.

Oil/Base Fraction (Fig. 8). Peaks 2, 3, 4, 5, 6, 7, and 9 in the 0-, 29-, and 60-day fractions and 1 and 8 in the 0-day and 15 in the 7-day fractions are due to plasticizer impurities. Lesser peaks, like 3.5, are also plasticizers. Peaks 10, 11, 12, 13, and 14 are due to trace amounts of alkylated pyridines that did not show up on the other fractions, most likely due to differences in extraction efficiency.

Fig. 6. Total Ion Chromatograms of the Neutral Fraction of 0-Day, 7-Day (37°C Incubated), and 29- and 60-Day (Refrigerator-Stored) Prequench-Tower Recycle Oil Sample

Fig. 7. Total Ion Chromatograms of the Acid Fraction of 0-Day, 7-Day (37°C Incubated), and 29- and 60-Day (Refrigerator-Stored) Prequench-Tower Recycle Oil Sample

Fig. 8. Total Ion Chromatograms of the Base Fraction of 0-Day, 7-Day (37°C Incubated), and 29- and 60-Day (Refrigerator-Stored) Prequench-Tower Recycle Oil Sample

Fig. 9. Total Ion Chromatograms of the Composite of Extracts Fraction of 0-Day, 7-Day (37°C Incubated), and 29- and 60-Day (Refrigerator-Stored) Prequench-Tower Recycle Oil Sample

Fig. 10. Total Ion Chromatograms of the Dried Oil Fraction of 0-Day, 7-Day (37°C Incubated), and 29- and 60-Day (Refrigerator-Stored) Prequench-Tower Recycle Oil Sample

4 CONCLUSIONS

As described in Secs. 1-3, the stability of organics in stored samples of coal gasification condensates was assessed by comparing total ion chromatograms of extracts and dried oil samples obtained from fresh and stored condensate samples. Except for minor differences in comparative total ion chromatogram traces that can be attributed to variations in the concentrations of extraneous plasticizers or to slight differences in column resolution or sample extraction, no significant changes in comparative total ion chromatogram traces were apparent during the storage periods for the dried oil, refrigerated, and incubated samples.

In light of the remarkable level of correspondence between chromatograms of specific fractions, it is concluded that those organics of the gasification condensates which are extractable by typical liquid-liquid extraction methods, and sufficiently volatile to pass through the capillary column into the mass spectrometer, are stable at refrigerator temperatures (2-4°C) for at least 60 days and at incubator temperature (37°C) for at least 7 days.

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This study could not have been completed at HYGAS without the support and assistance of the Institute of Gas Technology (IGT). Louis Anastasia of IGT assisted in obtaining suitable samples for study.

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The following individuals provided information on state-of-the-art methods for the storage of samples containing organic constituents similar to those found in coal gasification condensates: Ronald Atlas, University of Louisville; George Baughman, U.S. Environmental Protection Agency (EPA), Athens, Georgia; Clayton McAuliffe, Chevron Oil Company; Donald Mackay, University of Toronto; and John Kopp and Thomas Beller, U.S. EPA, Cincinnati.

APPENDIX

Sample Check-In, Coding, and Transfer Protocols
Used at Argonne National Laboratory

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Check-in and transfer control for *fresh* samples are handled at Argonne by Sally Vargo of the Energy and Environmental Systems Division (EES). Each new sample from a coal-gasification plant is examined by Ms. Vargo, who notes its condition and logs it in, using the log sheet shown in Fig. A.1. An identification number is then assigned to each sample and Ms. Vargo completes the check-in procedure by affixing to the sample container an "ANL/EES" sticker bearing the sample identification number, signing the log sheet, and placing the sample in a special refrigerator. (This refrigerator is dedicated to the storage of samples from coal gasification plants. It is locked and access to it is carefully controlled.) Each Friday, copies of new log sheets are sent to individual Argonne scientists who require the information. The master log sheets are kept in a locking file cabinet.

Transfer of portions of (or entire) *bulk* samples within or beyond the Argonne site requires communication with Ms. Vargo, who (1) permits access to the refrigerator where the samples are stored, and (2) enters the particulars of each transfer on the "EES Sample Transfer Log for Bulk Samples" (Fig. A.2). Ms. Vargo also keeps a record of bulk sample transfers after the samples leave EES; see for example Fig. A.2. This record permits tracing any given sample as it moves to its first work-up point in the Argonne system.

The typical flow of bulk samples is to Argonne's Analytical Chemistry Laboratory (ACL) for work-up into splits or fractions for analysis by scientists in EES and the Biological and Medical Research Division (BIM); see Fig. A.3.

Bulk samples receive identification numbers according to the following format:

ANL BULK SAMPLE LOG-IN RECORDSAMPLE I.D. NO. 72/H/G/O/1

Pilot Plant: HYGAS

Run No.: 72

Date Collection: 25 May 78

Time Collected: 1155 hr

Transport to ANL

Person: Sather

Time: 1430

Date: 25 May 78

Description: "Recycled Oil"
"Location: 4.06-42"
"Sample 1" = 1/2 gal.
"Sample 2" = 1/2 gal.

Special Comments:

500 psi run

Signature

Fig. A.1. Example of Log Sheet Used to Check-In a Bulk Sample from a Coal Gasification Pilot Plant

MASTER

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EES SAMPLE TRANSFER LOG

I.D. NO.	CONTAINER SIZE	DATE	TIME	TRANSPORTER	TO
72/H/G/W/2	1 GAL	25/V/78	1510	Sather	Bruce Hass
72/H/G/W/2	1 GAL	25/V/78	1510	Sather	Sid Bourne
72/H/G/O/1	1/16 GAL	25/V/78	1510	Sather	Bruce Hass
72/H/G/O/1	1/2 GAL	29/V/78	1130	Sather	Sid Bourne
72/H/G/O/1	1/16 GAL	31/V/78		Sather	Sid Bourne
72/H/G/W/2	1 GAL	31/V/78		Sather	Sid Bourne
72/H/G/O/1	1/16 GAL	2/VI/78		Sather	EES
62-64/H/WW/1	1 GAL	7/VI/78	1050	Sather	Sid Bourne
62-64/H/WW/2	1 GAL	7/VI/78	1050	Sather	Sid Bourne
72/H/G/O/1	1/2 GAL	7/VI/78	1050	Sather	Sid Bourne
72/H/G/O/3	1 GAL	7/VI/78	1050	Sather	Sid Bourne
72/H/G/W/2	1 GAL	22/VI/78	1600	Sather	Sid Bourne
72/H/G/O/1	1/16 GAL	22/VI/78	1600	Sather	Sid Bourne
62-64/H/WW/1	1 GAL	22/VI/78	1600	Sather	EES
62-64/H/WW/2	1 GAL	22/VI/78	1600	Sather	EES
72/H/P/W/4	9, 1 GAL	5/VII/78	2300	Sather	Sid Bourne
72/H/G/O/1	2, 1/2 GAL	5/VII/78	2300	Sather	EES
72/H/G/O/1	1/2 GAL	5/VII/78	2300	Sather	Bruce Hass
72/H/G/O/1	2, 1/2 GAL	5/VII/78	2300	Sather	EES
72/H/G/O/3	1/2 GAL	5/VII/78	2300	Sather	Bruce Hass
72/H/G/O/3	1/2 GAL	5/VII/78	2300	Sather	EES
72/H/G/W/2	5 GAL	5/VII/78	2300	Sather	Bruce Hass
72/H/G/W/2	5 GAL	5/VII/78	2300	Sather	EES
72/H/P/W/4	1 GAL	5/VII/78	2300	Sather	Bruce Hass
72/H/P/W/4	1 GAL	5/VII/78	2300	Sather	EES
72/H/G/O/1	1/16 GAL	5/VII/78	2300	Sather	EES

Fig. A.2. Example of Sample-Transfer Log for Bulk Samples

Fig. A3. Typical Flow of Bulk Samples and Sample Fractions at Argonne (EES = Energy and Environmental Systems Division, ACL = Analytical Chemistry Laboratory, CEN = Chemical Engineering Division, and BIM = Biological and Medical Research Division)

If the foregoing bulk sample was fractionated by the ACL, appropriate designations are added to the identification number, as follows:

72/H/G/W/2/ACL/1

First in a series of fractions worked-up by ACL

Specific information concerning each fraction, generated by the ACL for example, is logged into the ACL log book. These details are extracted and transmitted with the fractions when they are sent to EES or BIM (see Fig. A.3).

If, for example, EES runs several GC/MS analyses on fraction 72/H/G/W/2/ACL/1, it may add further information to the code (e.g., 72/H/G/W/2/ACL/1/EES/3) if desired. This is left to the discretion of the EES analyst, who may be satisfied with entering the analytical details of the GC/MS runs as "Run 1, Run 2", etc., in the log book of GC/MS runs.

The laboratory notebooks of the various investigators in BIM, EES, and the Chemical Engineering Division (CEN) are kept in secure locations when not in use. The following individuals are responsible for notebook security in the various divisions: Sidney Bourne (CEN), Bruce Hass (BIM), and Leo Raphaelian (EES).

A chain-of-custody sample follower (Fig. A.4) also is prepared for each new sample (including each bulk sample). This follower accompanies each sample as it is handled in various ways by different analysts. The follower will usually proceed all the way to the individual who incorporates relevant data for the sample into a report (see Fig. A.4). In any event, the last person to receive the follower is responsible for its safekeeping. Each person creating a new sample must initiate a new chain-of-custody follower. Previous followers relating to the new sample must be attached to the new follower.

The purpose of the follower is to indicate the chain of custody for a sample. Notations under the "Operation Performed" heading are brief descriptions of what was done to each sample. These notations in no way take the place of laboratory workbook entries.

CHAIN-OF-CUSTODY SAMPLE FOLLOWER^a

COAL GASIFICATION ORGANICS AND MUTAGENIC EFFECTS STUDY
 ARGONNE NATIONAL LABORATORY
 ARGONNE, ILLINOIS 60439

SAMPLE CODE: 72/H/G/O/1	SAMPLE TYPE: <i>Recycle</i> oil from location 4.06-42	VOLUME COLLECTED: 1 gal.	MEDIUM OR SOLVENT: neat
ORIGIN REFERENCE: IGT Transfer Doc. No. 2154	NO. CONTAINERS: 2 (1/2 gal. each)	VOLUME ANALYZED: 1/16 gal.	OTHER INFO: 500 psi run stored @ 2-4°C

Relinquished by:	Received by:	Time	Date ^b	Operation Performed (aliquot, std. addition, etc; or remarks [how stored, etc.])
S. Vargo EES	R. Boston CEN	2:30 p.m.	2 JUN 78	Filter and make 0.5 ml splits; store at 3°C
R. Boston CEN	J. Smith BIM	9:30 a.m.	8 JUN 78	Prepare for bioassay run 32-2 store at 3°C
J. Smith BIM	R. Johnson BIM	3:00 p.m.	12 JUN 78	Bioassay via strain 3421
R. Johnson BIM	T. Larson BIM	9:45 a.m.	23 JUN 78	Date interpretation; report to DOE via ANL/BIM-20/3.
T. Larson BIM	S. Vargo EES	10:15 a.m.	1 DEC 78	incorporate in central file

^aEarlier follower(s) for this sample, if any, must be attached to this sheet.

^bWrite out month

Fig. A.4. Chain-of-Custody Sample Follower

After the follower has been used by the individual writing a report, the form is returned to Ms. Vargo who maintains the central file for all forms.

The functioning of this system is monitored by Michael Agresta of Argonne's Quality Assurance Division (QAD). He receives copies of all pertinent forms (Figs. A.1-A.3) and reviews them for omissions or inconsistencies. He also reviews entries in each division's laboratory notebooks on a monthly basis. Any variations from outlined procedures that are noted by Mr. Agresta are brought to the attention of the project managers for resolution.

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