Leaf Chemistry, 1992-1993 (ACCP)

Summary:

As part of NASA's Accelerated Canopy Chemistry Program (ACCP) analyses were performed for the determination of carbon constituents and nitrogen content in fresh forest foliage. Samples were analyzed using a series of extractions that yielded different carbon constituents: non-polar, polar, cellulose and lignin. Nitrogen analyses were conducted using a standard combustion procedure. Approximately 1000 leaf samples were collected from 5 geographically distinct sites and were analyzed at the University of New Hampshire to ensure consistency in analysis. Results were used as a calibration set for Visible/NIR reflectance and the estimation of canopy carbon and nitrogen concentrations.

Citation:

Cite this data set as follows (citation revised on September 30, 2002):

Aber, J. D., and M. Martin. 1999. Leaf Chemistry, 1992-1993 (ACCP). [Leaf Chemistry, 1992-1993 (Accelerated Canopy Chemistry Program)]. Data set. Available on-line [http://www.daac.ornl.gov] from Oak Ridge National Laboratory Distributed Active Archive Center, Oak Ridge, Tennessee, U.S.A. doi:10.3334/ORNLDAAC/421.

Table of Contents:

1. Data Set Overview:

Data Set Identification:

LEAF CHEMISTRY, 1992-1993 (ACCP)

Data Set Introduction:

Includes wet chemical analysis for samples collected at Howland, ME; Blackhawk Island, WI; Harvard Forest, Petersham, MA; Jasper Ridge, Dunnigan, and Pleasant Grove, CA; and Gainesville, FL.

Objective/Purpose:

As part of NASA's Accelerated Canopy Chemistry Program we performed analyses for the determination of carbon constituents and nitrogen content in fresh forest foliage. Samples were analyzed using a series of extractions that yielded different carbon constituents: non-polar, polar, cellulose and lignin. Nitrogen analyses were conducted using a standard combustion procedure. Approximately 1000 samples were analyzed and used as a calibration set for Visible/NIR reflectance and the determination of plot level canopy chemistry.

Summary of Parameters:

Leaves from selected species were collected from defined plots and analyzed using a series of extractions that yielded different carbon constituents: non-polar, polar, cellulose and lignin. Carbon, hydrogen, and nitrogen analyses were conducted using a standard combustion procedure.

> 1. sample_id 2. site_id 3. plot_id 4. coll_date 5. lab 6. species 7. wet_ wgt 8. dry_wgt 9. nonpolar
> 10. polar 11. cellulose 12. lignin 13. carbon 14. hydrogen 15. nitrogen 16. leaf_wgt
> 17. water_thickness 18. chlorophyll_a 19. chlorophyll_b 20. lutein 21. Neoxanthin
> 22. beta_carotene

Discussion:

Standard tissues:

Three standard tissues (leaves of Quercus rubra, Robinia pseudoacacia and Acer saccharm) were collected in bulk for quality assurance/quality control by repeated carbon fractionation analysis. These species were selected to characterize the range in nitrogen and lignin concentrations associated with northern temperate forest canopies. The standard tissues were collected from three canopy levels (upper, mid and understory) to ensure a well mixed sample of both sunlit and shaded leaves.

Interlaboratory samples:

A total of 31 samples collected by NSF's Long Term Intersite Decomposition Experiment Team (LIDET) were analyzed for comparison of our methods with other labs. Sample types included deciduous and coniferous foliage, as well as roots and grasses collected from all major terrestrial biomes in North America.

Field samples:

Field samples from a number of sites were collected during the summer of 1992 within 10 days of the AVIRIS overflights. Green leaves were collected from five trees of each dominant overstory species on each plot sampled. Leaves were collected by shooting small branches from the canopy with a shotgun. Each sample was a composite of leaves collected from several heights in the canopy.

Chemical Analyses:

Sample preparation:

Prior to analysis, samples were oven dried at 70 degrees C for 48 hours, ground to 20-mesh and homogenised.

Proximate carbon fraction analysis:

Carbon constituent analyses were performed using a series of extractions modified from those used by Ryan, et. al. Two grams of material were refluxed with 75 ml of dichloromethane for five hours to remove the non-polar component of the sample (fats, waxes, and other organic soluble materials) using a block digestor and condenser apparatus modified from a TAPPI procedure. Polar extractives such as phenols, simple sugars, starch and simple amino acids were removed by boiling a subsample (1.6 g) of tissue from the non-polar analysis for three hours in deionised water and then filtering. Sample size was increased from the recommended 0.5 g to 1.6 g to ensure sufficient post polar sample quantity for the acid digestion. Lignin and cellulose content were determined by a modified wood-products chemistry procedure. A 0.5 g subsample of post polar material was digested in 72% sulpheric acid for 1 hour in a 30 degree C water bath. This was then diluted with water until a 28:1 water to acid ratio was formed to carry out secondary hydrolysis while boiling for 4 hours. To wet the sample thoroughly we used 1.5 ml of acid for each 100 mg of sample, a 0.5 ml increase in acid over the quantity Effland recommeded. At the end of this process the sample was filtered. Residual tissue was considered lignin and mass lost during digestion was considered cellulose. The percentages of the different constituents were determined gravimetrically. Oven-dried samples were temporarily stored in dessicators to prevent gaining moisture from the atmosphere before weighing.

Ash determination :

For each of the standard materials used in the ash determination procedure, a separate carbon fraction run was used to determine the amount of ash removed in each step. A block of 25

samples was used and subsamples were taken after each step of the analysis (post non-polar, post polar and post digest) and ashed at 500 degrees C for 5 hours to determine ash concentration.

Nitrogen analyses:

Nitrogen determination was performed using a Perkin-Elmer 2400 CHN Elemental Analyser. The CHN analyser combusts a sample to convert elements to gases (CO_2, H_2O, N_2). The resulting data are weight percentage of carbon, hydrogen and nitrogen (CHN). In addition, a total Kjeldahl nitrogen (TKN) determination was performed on a subset of 100 samples. This method uses sulphuric acid and a digestion catalyst of potassium sulphate, cupric sulphate and selenium powder on ground leaf samples to generate a liquid sample for colorimetric analysis, and was included as a alternate method of nitrogen determination included for QA/QC as assessment. In addition, three replicate samples of each of the three standard materials were sent to Oregon State University for a similar Kjeldahl analysis. Finally, three National Bureau of Standards (NBS) 1.2% nitrogen pine foliage standards were included in each of four Kjeldhal runs.

Fractional Ash Content:

Initial sample ash content for the three standard materials was 4.00%. Mean, SD, and CV for ash concentration were calculated after each step of the carbon fractionation procedure. Coefficient of variation for the post digest ash content for three of the six standard runs was greater than 100%. Low ash concentrations in the sample combined with very small sample size (less than 0.35 g post digest) made measurement of this fraction with any level of consistency and accuracy extremely difficult. Two of the six runs had calculated negative post digest ash content caused by the post-ash sample weighing more than the pre ash sample. A reasonable explanation for this is that the atmospheric water weight gain during handling and weighing for the post ash sample was greater than the weight of the combusted organic material. Based on these results we determined that the amount of error associated with attempting to apportion ash among the four fractions would only compound the error in an already complex analysis. Additionally, the results do not suggest that ash was conserved through the extraction procedure, which would be reflected in constantly increasing ash concentrations with each step. The most conservative assumption is that ash is removed in proportion with mass loss during the extraction steps, resulting in a constant ash concentration. Therefore, no ash correction was made to the mass determinations from the carbon fraction analyses.

Related Data Sets:

Calculated Leaf Carbon and Nitrogen, 1992 (ACCP) Visible and Near-Infrared Reflectance Spectra, 1992-1993 (ACCP) Site AVIRIS Images, 1992 (ACCP)

2. Investigator(s):

Investigator(s) Name and Title: Dr. John D. Aber, University of New Hampshire 03824

Title of Investigation:

Accelerated Canopy Chemistry Program

Contact Information:

Mary Martin, University of New Hampshire office: (603)862-1792, fax: (603)862-0188.

3. Theory of Measurements:

Percentages of different carbon constituents were determined by measuring mass loss at each step. Lignin and cellulose were determined using a 72% sulfuric acid digestion. Residual tissue from the digest was considered lignin and mass lost during the digestion was considered cellulose.

4. Equipment:

Sensor/Instrument Description:

Weighing balance. CHN elemental analyzer.

Collection Environment:

Closed canopy forests:

Source/Platform:

Laboratory.

Source/Platform Mission Objectives:

Analyses were performed in a controlled laboratory setting.

Key Variables:

Leaf carbon fractions including nonpolar and polar extracts, cellulose, and lignin. Elemental carbon, nitrogen, and hydrogen content of leaf samples. Leaf pigments were measured only at one site.

Principles of Operation:

Carbon fractions were measured using a sequential extraction technique and determined gravimetrically. The CHN analyzer combusts a sample to convert elements to gases (CO_2, H_2O, N_2). The resulting data are weight percentage of carbon, hydrogen and nitrogen (CHN).

Sensor/Instrument Measurement Geometry:

not applicable

Manufacturer of Sensor/Instrument:

Perkin-Elmer 2400 CHN Elemental Analyser: Perkin-Elmer Corp. Oak Brook, IL

Calibration:

Specifications:

not applicable

Tolerance:

not applicable

Frequency of Calibration:

not applicable

Other Calibration Information:

not applicable

5. Data Acquisition Methods:

Sample Collection:

Field samples were collected within 10 days of the AVIRIS overflights. Green leaves were collected from five trees of each dominant overstory species on each plot sampled. Leaves were collected by shooting small branches from the canopy with a shotgun. Each sample is a composite of leaves from several heights in the canopy. For needle-leaved samples, no separation was made between needles of different ages. Foliage was removed from the sampled branches and put in ziplok bags.

Sample Processing:

The sample+bag weight was recorded and then a mean bag weight was subtracted to derive a fresh green weight for the sample within several hours of collection. The samples were then placed in paper bags and oven dried at 70 C for 48 hours. Water content of the samples were determined by the weight difference between fresh and oven dried weights. After drying, the leaves were ground with a Wiley Mill to pass through a 1mm mesh screen.

Wet Chemistry Analyses:

Dried and ground leaves were analyzed for chemical composition. Cellulose and lignin were measured using a sequential extraction/digest method (Newman94). This method separates carbon compounds into nonpolar extractives (fats, waxes), polar extractives (starches, sugars, simple amino acids and polyphenols), acid digestible (cellulose), and acid insoluble (lignin) fractions. The percentages of the different constituents were determined gravimetrically. The CHN combustion method (Perkin-Elmer 2400) was used to determine carbon, nitrogen and hydrogen content of each sample.

6. Observations:

Data Notes:

Leaf samples were taken at all sites within ten days of the AVIRIS overflights.

Field Notes:

7. Data Description:

Spatial Characteristics:

Spatial Coverage:

Harvard Forest, Petersham, MA, latitude 42.4950, longitude -71.7981

Blackhawk Island, WI, latitude 43.6333, longitude -89.7583

Howland, ME, latitude 45.2222, longitude -68.7356

Gainesville, FL, latitude 29.7000, longitude -82.1667

Jasper Ridge, CA, latitude 37.4111, longitude -121.7631

Dunnigan, CA, latitude 38.9167, longitude -120.1122

Pleasant Grove, CA, latitude 38.7292, longitude -120.4581

Spatial Coverage Map:

not applicable

Spatial Resolution:

not applicable

Projection:

not applicable

Grid Description:

not applicable

Temporal Characteristics:

Temporal Coverage:

Summer and early fall 1992.

Temporal Coverage Map:

not applicable

Temporal Resolution:

not applicable

Data Characteristics:

Parameter/Variable:

Please note that minimum and maximum values were determined across all data set granules.

1.
variable=sample_id
definition=unique sample identifier that can be associated with an individual spectrum in related data set

2.
variable=site_id
definition=alphanumeric code for the sampling site
code=bhi: Blackhawk Island, WI
code=how: Howland, ME
code=hf: Harvard Forest, Petersham, MA
code=gain: Gainesville, FL
code=jr: Jasper Ridge, CA
code= rice: Dunnigan and Pleasant Grove, CA

3.
variable=plot_id
definition=plot number that can be associated with plot chemistry values in related data set

4.
variable=coll_date
definition=date field samples were collected
units=YYMMDD (format)
minimum=920618
maximum=930527

5.
variable=lab
definition=laboratory where analyses were performed
code=UNH: University of New Hampshire (CSRC)
code=UCD: University of California at Davis

6.
variable=species
definition=coniferous and deciduous forest species collected
code=see companion file for LTER codes

7.
variable=wet_ wgt
definition=fresh/wet sample weight
units=g
minimum=6.0
maximum=201.1

8.
variable=dry_wgt
definition=dry sample weight after 48 hours at 70oC
units=g
minimum=0.77
maximum=84.31

9.
variable=npolar
definition=fats, waxes, and other soluble organic compounds
units=% of wet weight
minimum=1.00
maximum=14.07

10.
variable=polar
definition=phenols, simple sugars, starchs, and simple amino acids
units=% of wet weight
minimum=9.91
maximum=55.78

11.
variable=cellulose
definition=mass lost during acid digestion procedure
units=% of wet weight
minimum=23.69
maximum=67.57

12.
variable=lignin
definition=acid insoluble material
units=% of wet weight
minimum=11.62
maximum=33.70

13.
variable=carbon
definition=carbon
units=% of dry weight
minimum=39.09
maximum=54.37

14.
variable=hydrogen
definition=hydrogen
units=% of dry weight
minimum=5.64
maximum=7.25

15.
variable=nitrogen
definition=nitrogen
units=% of dry weight
minimum=0.54
maximum=5.25

16.
variable=leaf_wgt
definition=leaf weight
units= [mg dry leaf][cm^-2 leaf)]
minimum=3.090
maximum=25.5887

17.
variable=water_thickness
definition=average water thickness
units= [g water][cm^-2 leaf]

minimum=0.0053
maximum=0.0552

18.
variable=chloro_a
definition=chlorophyll_a
units= [mg][g^-1 dry weight]
minimum=0.8330
maximum=13.3710

19.
variable=chloro_b
definition=chlorophyll_ab
units= [mg][g^-1 dry weight]
minimum=0.3589
maximum=5.2283

20.
variable=lutein
definition=lutein
units= [mg][g^-1 dry weight]
minimum=0.3893
maximum=2.0760

21.
variable=neoxanthin
definition=neoxanthin
units= [mg][g^-1 dry weight]
minimum=0.0804
maximum=0.4770

22.
variable=beta_car
definition=beta carotene
units= [mg][g^-1 dry weight]
minimum=0.1213
maximum=1.2698

Sample Data Record: Applicable for all data files except Jasper Ridge, CA.

```
sample_id site_id plot_id collection date lab species fresh weight
```

```
92BHIS10BW1    BHI      10        920627        UNH    TIAM       34.2
dry weight non-polar polar cellulose lignin carbon hydrogen nitrogen
```

```
 11.15 2.53 30.96 42.23 24.28 47.13 6.32 3.28
```

Applicable for Jasper Ridge, CA data file.

```
sample_id site_id coll_date lab species leaf_weight water_thickness
```

```
   JR11       JR      920630    UCD/UNH    ERCA6      25.5887         0.0261
```
```
cellulose lignin carbon nitrogen chlorophyll_a chlorophyll_b lutein
```

```
 38.61      14.83   49.79    1.05        1.8331          0.7918      0.5286
neoxanthin beta_carotene
```

```
 0.0983 0.4288
```

8. Data Organization:

Data Granularity:

The following files contain carbon fractionation and nitrogen content data for leaf samples collected at ACCP study sites. This is really only one data set, but with multiple granules, that is, a data file for each site. This allows convenient matching of site leaf chemistry data with the leaf spectra in the related data sets.

bhi_chem.dat
how_chem.dat
hf_chem.dat
gainchem.dat
jr_chem.dat
ricechem.dat

A general description of data granularity as it applies to the IMS appears in the EOSDIS Glossary.

Data Format:

The data files are ASCII files. The first two lines are metadata. The first line contains the filename and the number of data records to follow. The second line contains the comma delimited column headings. The data values are also comma delimited. Missing values are denoted by -999.

Applicable for all data files except Jasper Ridge, CA.

bhi_chem.dat, Xxxxx records

```
sample_id,site_id,plot_id,coll_date,lab,species,fresh_wgt,dry_wgt,npolar,polar,cellulose,lignin,carbon,hydrogen,nitrogen
```

```
92BHIS10BW1,BHI,10,920627,UNH,TIAM,34.2,11.15,2.53,30.96,42.23,24.28,47.13,6.32,3.28
```

```
92BHIS10BW2,BHI,10,920627,UNH,TIAM,22.6,7.72,3.14,31.34,43.09,22.43,47.72,6.39,3.51
```

Applicable for Jasper Ridge, CA data file.

```
jr_chem.dat, xxxxx records

sample_id,site_id,coll_date,lab,species,leaf_wgt,water_th,cell,lignin,carbon,nitrogen,chloro_a,chloro_b
,lutein,neoxanth,beta_car

JR11,JR,920630,UCD/UNH,ERCA6,25.5887,0.0261,38.61,14.83,49.79,1.05,1.8331,0.7918,0.5286,0.0983,0.4288

JR13,JR,920630,UCD/UNH,ACMA,5.5666,0.0075,34.8,20.77,47.64,1.96,8.4887,2.7765,1.4056,0.3431,0.8424
```

9. Data Manipulations:

Formulae:

Derivation Techniques and Algorithms:

not applicable

Data Processing Sequence:

Processing Steps:

not applicable

Processing Changes:

not applicable

Calculations:

Special Corrections/Adjustments:

not applicable

Calculated Variables:

The following are the formulas used to calculate carbon constituents:

% non polar = (Pre non polar mass - post non polar mass)/ pre non polar mass; % polar = ((pre polar mass - post polar mass)/ pre polar mass) * (1-% non polar); % cellulose = ((pre digest mass - post digest mass)/ pre digest mass) * (1- % non polar - % polar); and % lignin = (post digest mass / pre digest mass) * (1- % non polar- % polar).

Graphs and Plots:

not applicable

10. Errors:

Sources of Error:

Quality Assessment:

Data Validation by Source:

not applicable

Confidence Level/Accuracy Judgment:

Samples were analyzed using a "block" digestion apparatus that processed 25 samples per batch. Within each block, two standard samples and one duplicate were included to analyze individual block chemistry as well as the consistency of that blocks results (chemistry) with the other runs throughout the ACCP project. The mean, standard deviation (SD) and coefficient of variation (CV) of the two standards and duplicate samples were calculated as a measure of interblock chemistry. Any runs with greater than 10% CV for duplicates or standards for any constituent were reanalyzed. The standards in each run were also compared to the mean constituent values from the initial analyses. If any constituent values of the current batch standards deviated from the initial mean by more than 10%, the entire run was reanalyzed. This assured no greater than 10% error between individual data runs and the standard analyses carried out at the beginning of the project. A few batches had to be reanalyzed (approximately 5 out of 60).

Measurement Error for Parameters:

not applicable

Additional Quality Assessments:

not applicable

Data Verification by Data Center:

not applicable

11. Notes:

Limitations of the Data:

No known limitations of the data

Known Problems with the Data:

No known problems with the data

Usage Guidance:

not applicable

Any Other Relevant Information about the Study:

A supplemental file of leaf chemistry data collected prior to ACCP is included as a companion data set. Check GRNLEAF.dat for results of analyses on additional tree species. The samples came from various New England sites, primarily Durham, NH, Harvard Forest, MA, and Mt. Ascutney, VT.

12. Application of the Data Set:

These wet chemical analyses are a key portion in the ACCP project goals. These data were used to develop a green foliage calibration for an NIRS 6500.

13. Future Modifications and Plans:

not applicable

14. Software:

Software Description:

not applicable

Software Access:

not applicable

15. Data Access:

Contact Information:

ORNL DAAC User Services Oak Ridge National Laboratory
Telephone: (865) 241-3952
FAX: (865) 574-4665 Email: ornldaac@ornl.gov

Data Center Identification:

ORNL Distributed Active Archive Center Oak Ridge National Laboratory
Telephone: (865) 241-3952
FAX: (865) 574-4665 Email: ornldaac@ornl.gov

Procedures for Obtaining Data:
Users may place requests by telephone, electronic mail, or FAX. Data is also available via the
World Wide Web at http://daac.ornl.gov.

Data Center Status/Plans:

These data are available from the ORNL DAAC. Please contact the ORNL DAAC User Services
Office for the most current information about these data.

16. Output Products and Availability:

Available via FTP or on CD-ROM. A complete listing of all data sets can be found on the World
Wide Web at http://daac.ornl.gov.

17. References:

Newman, S. D.,M. E. Soulia, J. D. Aber, B. Dewey, and A. Ricca. 1994. Analyses of forest
foliage I: Laboratory procedures for proximate carbon fractionation and nitrogen determination.
Journal of Near Infrared Spectroscopy 2:5-14.

18. Glossary of Terms:

A glossary is available at http://cdiac.esd.ornl.gov/cdiac/glossary.html. For additional terms, see
the EOSDIS glossary at
http://www-v0ims.gsfc.nasa.gov/v0ims/glossary.of.terms.html.

19. List of Acronyms:

ACCP

Accelerated Canopy Chemistry Program

URL

Uniform Resource Locator

20. Document Information:

March 29, 1999 (citation revised on September 30, 2002)

Document Review Date:

March 29, 1999

Document URL:

http://daac.ornl.gov