

FEED AND WATER SAMPLING FOR ANALYSIS

B. I. Agag

Biochemistry and Nutritional Deficiency Diseases Department, Animal Health Research Institute, Agricultural Research Center

1. FEED SAMPLING:

The most important step in obtaining meaningful information about feed quality is the collection of a representative sample. The feed sample submitted to laboratory for analysis should be indicative of what is present in the entire hay mow, grain bin, ration store or silo. The following are general guidelines to follow when collecting feed samples.

1.1-Sampling dry feeds:

Dry feeds are feeds with 12% moisture or less. For example, individual ingredients (grains, soybean meal, fish meal, meat meal, bone meal), total mixed ration (concentrate ration), supplements and forages.

1.1.1-Sampling ingredients or mixed ration:

- Collect 10-15 random hand grab samples from an entire delivery or a bulk feed bin (Equine Research Foundation, 2002) and
- Collect random deep probe samples from bagged feed. The probe is inserted diagonally and as horizontally as possible from one corner of the bag to the other (Official Methods of Analysis, 2000).
- In lots of 1-10 bags, all bags are sampled. In lots of 10-100 bags, 10 bags are sampled (Hongsuway, 2001). In lots more than 100 bags, square root of total number is sampled (Table, 1). Take one core from each bag to be sampled, except that for lots of 1-4 bags take about 5 diagonal cores from each bag (Official Methods of Analysis, 2000).

- Pool subsamples, mix thoroughly and then reduce the size by making a cone of sample and quartering (Hongsuwong, 2001) to about 1 kg composite sample (Fig., 1). [Pour the entire sample into a conical pile (1) on a clean paper or plastic. Then divided the sample into four equal parts or quarters (2), saving the opposite two quarters (3). If the sample is still too large, repeat the procedure until the proper sample size of 1 to 1½ quarts is obtained].

- Place composite samples in a double thickness either paper or cotton bags for storage or submitting to the laboratory (Tarr, 2002).

1.1.2-Hay sampling:

1.1.2.1-Baled hay (bales):

- Select 10-20 bales at random from each lot and collect one sample from each bale by using a forage sampler (core sampler) or probe at least 12-18 inches long and $\frac{3}{8}$ inch internal diameter. For rectangular bales, sample from the end and for round bales, sample across the bale at the center.

- Collect bales at random and do not collect samples with hand or use flakes of hay. Hand sampling may provide unreliable results and flakes may not even represent the bale. If hand sampling is used, make appropriate combination of leaves and stems (Schroeder and Sedivee, 1993).

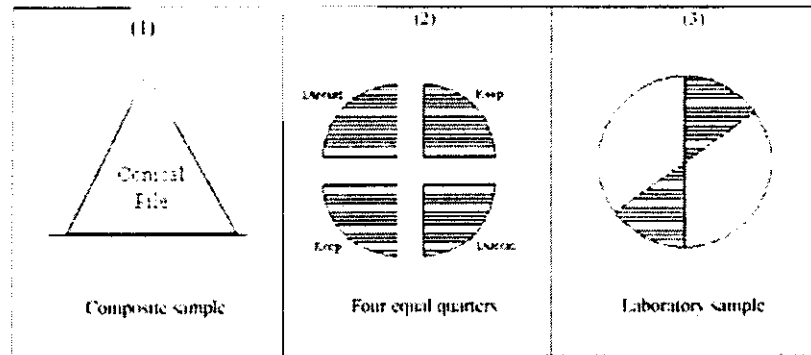


Fig. (1): Reducing size of composite samples by coning and quartering

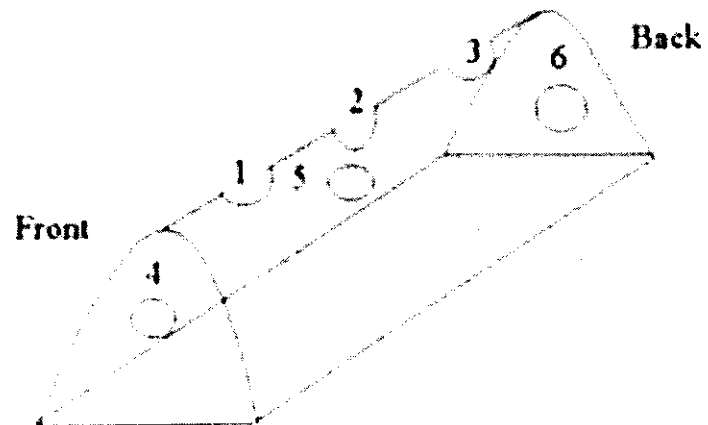


Fig. (2): Sampling compressed hay stacks

Table (1) : Sampling scheme of consignments of more than 100 bags (N = number of bags in consignment, n = number of bags to be sampled).

N	n	N	n	N	n
101 - 121	11	1601 - 1681	41	4901 - 5041	71
122 - 144	12	1682 - 1764	42	5042 - 5184	72
145 - 169	13	1765 - 1849	43	5185 - 5329	73
170 - 196	14	1850 - 1936	44	5330 - 5476	74
197 - 225	15	1937 - 2025	45	5477 - 5625	75
226 - 256	16	2026 - 2116	46	5626 - 5776	76
257 - 289	17	2117 - 2209	47	5777 - 5929	77
190 - 324	18	2210 - 2304	48	5930 - 6084	78
325 - 361	19	2305 - 2401	49	6085 - 6241	79
362 - 400	20	2402 - 2500	50	6242 - 6400	80
-	-	-	-	-	-
-	-	-	-	-	-
-	-	-	-	-	-
-	-	-	-	-	-
1226 - 1296	36	4226 - 4356	66	9026 - 9216	96
1297 - 1369	37	4357 - 4489	67	9217 - 9409	97
1370 - 1444	38	4490 - 4624	68	9410 - 9604	98
1445 - 1521	39	4625 - 4761	69	9605 - 9801	99
1522 - 1600	40	4762 - 4900	70	9802 - 10000S	100
N = more than 10.000			N = square root of N		

Table (2) : Sample container, preservative and maximum holding time (dead time analysis) for a variety of parameters (Environmental Protection Agency, 1983; Government du Quebec, 2001 and Dryden Aqua Limited, 2002)

Determination	Sample container	Preservatives	Holding time
pH	P, G	None, cool to 4°C	Immediately
Color	G	None, cool to 4°C	48 hours
Odor	G	None, cool to 4°C	24 hours
Oxygen, dissolved	G	None	Immediately
Ammonia	P,G (G)	H ₂ SO ₄ to pH <2, cool to 4°C	28 days
Nitrate and nitrite	P,G (G)	None, cool to 4°C	48 hours
Nitrate and nitrite	P,G (G)	H ₂ SO ₄ , cool to 4°C	28 days
Phosphates	P, G	None, cool to 4°C	48 hours
Sulphates	P, G	None, cool to 4°C	48 hours
Alkalinity	P, G	None, cool to 4°C	14 days
Hardness total (CaCO ₃)	P,G (G)	NH ₃ to pH < 2	6 months
Total solids (TS)	P, G	None, cool to 4°C	7 days
Total dissolved solids (TDS)	P, G	None, cool to 4°C	7 days
Total suspended solids (TSS)	P, G	None, cool to 4°C	7 days
Cyanide, total (CN)	P,G (G)	NaOH to pH > 12	days 14
Phenols	G	H ₂ SO ₄ to pH < 2	28 days

1.1.2.2- Loose or compressed hay stacks:

- Use a forage sampler or a hay probe at least 24 inches long to collect samples (Anderson, *et al.*, 1995).
- Collect 15 or more samples from each lot.
- Sample loose hay stacks from the top and from the stack sides of the mow, or place the hay sampler beneath your feet to compress the hay and sample between your feet (Schroeder and Sedivee, 1993).
- You can sample loose hay without a forage sampler by removing a handful of hay from many (15-20) positions from each lot of hay. Be careful to save all leafy material belonging to the sample.
- Compress loaf stacks, require six sampling locations (Anderson *et al.*, 1995). These locations are: 1. Top front, Top middle, 3. Top rear, 4. Lower front side, 5. Lower middle side, and 6. Lower rear side (Fig., 2).
- Mix subsamples of baled hay or hay stack well and take a 500 gm composite sample, using the quartering procedure.
- Place samples into clean double layer paper or cotton bags and seal tightly (Tarr, 2002).

Nota bene:

- A lot of hay should represent hay harvested from the same cutting and the same stage of maturity (Hannaway and Ballersted, 2001).
- Use stainless steel sampler to avoid contamination of the sample.
- Use clean disposable glove for each feed sample to prevent contamination.

1.2- Sampling wet feeds:

Wet feeds are any feeds with 15% moisture or greater e.g. corn silage, haylage and high moisture grains. Feeds between 2 and 15% moisture is safer to handle as a wet feeds. For sampling wet feeds follow these steps:

- Take 8-12 samples of wet feeds at each of 3-5 feedings or feed removal from the storage (Tarr, 2002), or
- Take a handful of silage from 10-12 random depths and locations within the upright silo, and remove a column about 6 inches by 12 inches wide on the open end of the horizontal silo (Anderson *et al.*, 1995).
- Mix samples thoroughly in a bucket and take a 750-1000 gm composite sample.
- Place composite sample in a thick double plastic bag, squeeze out most of the air and seal it tightly (Schroeder and Sedivee, 1993; Tarr, 2002).

1.3-Sample handling:

Proper handling of the feed sample between the farm and laboratory, ensures best results. General rules are:

- Label or identify all samples using a permanent marker (Equine Research Foundation, 2002) with : (Farm or store name, Sample number, Supplier's name, Address, Telephone number, Ingredients or feed name, Production code or date of delivery, if no code, Tests required and Who should receive a copy of the results other than the sender).
- All forages should be chopped to make handling easier (Schroeder and Sedivee, 1993).
- Avoid direct sunlight and damage to bags (Anderson *et al.*, 1995).
 - Send samples to the laboratory as quickly as possible or store dry samples in cool dry place and freeze wet samples till they are submitted for analysis (Anderson *et al.*, 1995, Tarr, 2002).
 - Send samples in good containers, as keeping wet samples in cool containers could guarantee a minimum of a silage like fermentation (Anderson *et al.*, 1995).

1.4-Laboratory sample preparation:

Laboratory sample preparation is the process of converting the sample

received at the laboratory into a homogenous material suitable for analysis. This process generally involves drying and/or grinding (Undersander *et al.*, 1993).

1.4.1- Preparing samples greater than approximately 85% dry matter:

- Chop samples of whole plants into about ½ inch pieces.
- Grind entire sample to pass 4-6 mm sieve. If the sample is wet to grind (less than 9% dry matter) dry the sample using hot air oven.
- Reduce the sample size to amount desired for laboratory subsample (by making a cone of sample and quartering). Transfer the remainder to a plastic bag, seal and label. Grind the reduced sample to fineness desired for analysis. Thoroughly mix the ground sample, transfer it to an airtight container and label immediately.

1.4.2- Preparing samples less than approximately 85% dry matter:

- Chop samples of whole plants into about ½ inch pieces, place on clean plastic sheet and mix thoroughly.
- If the entire sample can not be dried, reduce the sample size.
- Dry the reduced sample and grind it to fineness desired for analysis.
- Thoroughly mix the ground sample, transfer it to an airtight container and label.

2- WATER SAMPLING PROTOCOL

2.1- Preparation of sample container:

Sample container or bottle must be suitable for sampling the water without affecting the compound or compounds that sample is to be analyzed for (Peterson, 2002). Containers made of either from plastic or pyrex glass are used. Plastic containers either high-density polyethylene or polypropylene might be preferable to glass from a

practical stand point because they will better withstand breakage (Environmental Protection Agency, 2002b).

The following procedures should be used when preparing all sample containers and glassware for monitoring pH, total solids, turbidity and total alkalinity of water (Environmental Protection Agency, 2002a):

- Wash each sample container with a brush and phosphate-free detergent.
- Rinse three times with tap water.
- Rinse three times with distilled or bidistilled water (Arizona State University, 2001).

The acid wash procedure should be used when preparing containers and glass ware for monitoring phosphorus, phosphates and nitrates because phosphorus molecules have a tendency to adsorb (attach) to the inside surface of containers (Environmental Protection Agency, 2002b). For acid washing, soak glass or polyethylene bottles and caps in 10% HCl for several hours or overnight and rinse thoroughly with water followed by distilled water. Dry in oven and cover or wrap with aluminum foil (Sheldon and Wiebe, 1997). If acid washing is impossible, rinse all glass ware and fill all vessels with distilled water only (Arizona State University, 2001).

Nota bene:

To minimize contamination and ensure sample integrity, basic precautions are necessary. These precautions include:

- Do not use metallic caps (German, 2002).
- Do not use containers of unknown origin to store samples.
- Samples collected for bacteriological examination should be taken in suitable sterile containers (Missouri University, 1995).

- Do not use soap or detergent for washing of sample containers without testing as many contain ammonia, phosphates, etc (Sheldon and Wiebe, 1997).
- Discard any bottle appearing stained on the inside or cracked (Arizona State University, 2001).

2.2- Sampling procedures:

Different procedures are used for water sampling according to its source (surface, well or tap water).

2.2.1. Sampling of surface water (River Nile, Canals, Ditches, Ponds and Lakes):

- Rinse sample bottle and cap three times with surface water at the sampling site. Be sure that do not put your fingers inside the bottle or the cap and to empty your rinse water away from the sampling location.
- Lower the open bottle upside down to arm's length.
- Under water, turn bottle upside right and let it fill up
- When no more bubbles come up, cap the bottle under water and bring back up to the surface (Walk, 2002).
- Label the bottle with the sample number. Complete the sample identification and submit the sheet with the sample. The reporting sheet should include:- Source and site of sampling , Collection time and date, Sample number assigned, Collector's name, Type of preservative added, Legal description (required for livestock drinking), Analysis requested, Any water quality problems (color, odor, etc., livestock illness or deaths and any other interesting or significant observations), Address, Phone No., Fax No. and E-mail address (German, 2002; Public Health Laboratory, 1997-2001).

Nota bene

- Take the sample away from the shore and avoid algae, soil and other foreign materials.
- Composite sample is obtained by mixing equal volumes of dip samples (collected at one point at regular time intervals or collected from multiple points such as varying depths, (Province of British Columbia, 1993b). Fill the composite sample bottle completely, because half filled bottle allow nitrogen bacteria to fix nitrogen from the air. In case of freezing of the sample, fill the bottle 90% full to allow for water expansion (German, 2002).
- Sample requiring filtration and/or preservation should be dealt with as soon as possible (Province of British Columbia, 1993b).

2.2.2- Sampling of well water:

- When sampling monitoring wells, begin with the one expected to have the best water quality and end with the one expected to have the worst water quality and proceed as follows (Minnesota Department of Agriculture, 2002):
- Rinse each bottle and its cap three times with water from the well before filling. To rinse, fill the bottle approximately one-third full, place cap and shake vigorously. Discard the rinse water after each rinse cycle.
 - Fill the bottle completely from the well.
 - Add any required preservative (filter first, if required), place the bottle cap and the label.
 - Record all field observations before proceeding to the next well.

2.2.3- Sampling of tap water (Public Health Laboratory, 1997-2002):

- Select taps (faucets) that are in frequent use, avoid any tap that is dusty, dirty or corroded.

- Allow the selected tap flow long enough (5 minutes), for water to arrive from the water main itself.
- Open the bottle, hold the bottle in one hand and the cap in the other (do not lay the bottle cap down or put it in your pocket, keep your fingers out of the bottle and hold the bottle under the tap, so no part of the tap will touch the inside of the bottle).
- Rinse the bottle and the cap 3 times with the tap water.
- Fill the sample bottle to the top (be careful that splashing drops of water from the ground or elsewhere do not get into the bottle or onto the cap).
- Cap the bottle immediately after filling and then turn off the tap.
- Add the sample label.

2.3- Processing the water sample (Dryden Aqua Limited, 2002):

Many samples need to be filtered before testing. In some cases the filtering steps must be done in the field as soon as the sample has been collected. Other samples require preservation (Table, 2). To stop any change as a result of physical, chemical or biological reactions that may take place between the time of sampling and the analysis. Changes may occur due to:

- Consumption of certain constituents by bacteria, algae, etc.
- Certain compounds being oxidized by the dissolved oxygen in the sample.
- Precipitation from the liquid e.g. calcium carbonate and aluminum hydroxide.
- Absorption of carbon dioxide from the air, changing the pH value.
- Adsorption of metals and certain organic compounds on the container surface.

These changes will be affected by the storage temperature, exposure to light, the nature of the container used and the time between sampling and analysis.

Fortunately, preservatives are available to prevent these changes.

Also it must be borne in mind the following precautions to minimize samples contamination and ensure sample integrity.

- Do not smoke while taking samples.
- Fill containers to the brim and stopper them tightly so that no air is left above the sample.
- Use an appropriate clean containers. Brown bottles should be used since this will reduce photosensitive reactions to a considerable extent.
- Keep samples at a temperature below that at the time of filling. Cooling between 2 and 5 degrees centigrade is adequate.
- Filter or centrifuge the sample immediately on receipt at the laboratory to remove suspended matter, sediment, algae and other microorganisms.

2.4- Sample transport to the laboratory for analysis:

Samples must be sent to the laboratory without delay (within 24 hours of sampling). This need to be done under appropriate conditions, often in a dark, clean, cooler with ice packs (Province of British Columbia, 1993a). If it is not possible to send the sample to the laboratory immediately after collection, refrigerate until it is sent.

REFERENCES

- Andreson, B.; T. Mader and R. Grant (1995). Sampling feeds for analysis File G331 Under: Dairy A-2, Feeding and Nutrition, pp.1-4. Electronic version issued March 1996. University of Nibraska. Pubs@unl.edu.
- Arizona State University (2001). Water sampling protocol. Center for Environmental studies, Page updated 27/2/2001, by CDZ.

- Dryden Aqua Ltd, (2002). Handling and preservation of water samples.PP.1-3. Butler field, Bonnyrigg, Edinburgh, Scotland
- Environmental Protection Agency (1983). Sample preservation. pp. XV-XX, Ohio, USA.
- Environmental Protection Agency (2002a). Water quality conditions. Office of water, EPA. Page revised 9/1/2002.
- Environmental Protection Agency (2002b). 5.6.Phosphorus.EPA, Office of water, EPA. Paper revised 9/1/2002.
- Equine Research Foundation (2002). Mare reproductive loss syndrome. Sampling recommendation for horse feed and grain. University Science Home Page. College of Agriculture. UK.
- German D.R. (2002). Sample identification and information form. Water Quality Laboratory. South Dakota State University.
- Government du Quebec (2001). Methods for taking and preserving samples for the application of the regulation respecting the quality of drinking water. The Minister Programs and services.
- Hannaway, D.B. and P.J. Ballerstedt. (1988). Testing the quality of Alfalfa Hay. Forage information system. Universities of Oregon, Washington and Idaho. Paper last updated October, 8, 1996.
- Hongsuwong, T. (2001). Sampling, sample handling and preparation in grains and cereals.PP.1-17.State Supervision Agency for Public Health. Netherland.
- Minnesota Department of Agriculture (2002). Ground water sampling guidance.PP.1-6.MD home State of Minnesota.
- Missouri University (1995). Water testing: What to test for. Water Quality Initiative PublicationWG100.PP.1-11.Missouri University, Columbia
- Official Methods of Analysis (2000). Association of Official Analytical Chemists.17th Ed., AOAC International, USA.
- Peterson, H.G. (2002). Water sampling, analysis and interpretation. Safe drinking water foundation, Canada
- Province of British Columbia (1997a). 3- Quality assurance/quality control. Published by the Resources Inventory Committee.
- Province of British Columbia (1997b). 4- Collecting samples. Published by the Resources Inventory Committee.
- Public Health Laboratory (1997-2002). Water sampling Instructions.PP.1-4. Placer Country, California.
- Schroeder, J.W. and Sedivee, K. (1993). Sampling feed for analysis. North Dakota State University, NDSU Extension Service.
- Sheldon,J.E. and Wiebe,W.J. (1997). Nutrient analysis methods manual.PP.1-3.Georgia River LMER Data.
- Tarr, B. (2002). Sampling feed to test for mycotoxins. Livestock@omarfa.gov.on.ca. Queens Printer for Ontario. Last updated : January 7, 2002.
- Undersander, D.; Mertens, D.R. and Thiex, N. (1993). "Laboratory sample preparation." National Forage Testing Association, Ohama, NE 68137.
- Walk, M.F.(2001). pH and Alkalinity. Sampling method and analysis protocol.PP.1-8.Mass WWP,MWWP home.

جمع عينات الأعلاف والماء بغرض التحليل المعمل

بدير ابراهيم عجاج

قسم بحوث الكيمياء الحيوية والنقص الغذائي و السموم - معهد بحوث صحة الحيوان بالسدي - مركز
البحوث الزراعية

في هذا الموضوع تم استبيان الطرق المعملية المتبعة لجمع عينات كل من الأعلاف والماء المستخدمة في تغذية الحيواني و شرب الحيوانات و الدواجن ، وذلك بغرض تحليلها معمليا لاثبات مدى صلاحيتها للاستهلاك والداجن من عدمه . وقد روعي في ذلك اختيار أبسط واحداث الطرق المستخدمة لهذا الغرض و التي تتفق وامكاناتنا الحقلية و الموثقة من قبل المنظمات والهيئات العالمية.
اولا: عينات الاعلاف:

تم عرض الطرق المستخدمة في جمع عينات الاعلاف المختلفة مثل:

١-الاعلاف الجافة: أي التي تحتوي علي نسبة رطوبة أقل من ١٥% ، و تشمل الأعلاف المركزة ومكوناتها مثل الذرة وفول الصويا ومساحيق السمك و اللحم و العظم ، وكذلك الاعلاف الجافة مثل القش و البرسيم المجفف والموجودة في صورة بالات مضغوطة أو أكوام غير مضغوطة .
٢- الأعلاف غير الجافة: أي التي تحتوي علي نسبة رطوبة أكثر من ١٥ % مثل السيلاج والنباتات والحبوب الخضراء . ومن خلال عرض الطرق الخاصة بجميع العينات السابقة تم توضيح كميات العينات الممثلة ، والأدوات المستخدمة في جمعها ، و العيوب المستخدمة في حفظها ،وكيفية نقلها الي المعمل و كذلك تجهيزها لاجراء التحاليل اللازمة .

ثانيا : عينات الماء :

تم عرض الطرق المختلفة لجمع عينات الماء من المصادر الآتية :

١- الماء السطحي : (المجري المائي) مثل القنوات والترع و نهر النيل والبرك والمستنقعات والبحيرات
٢- الآبار والينابيع .

٣- صنابير المياه . و قد تم توضيح النقاط الآتية أثناء جمع عينات الماء :

تحديد طرق اختيار وتجهيز عيوب أ و زجاجات جمع عينات الماء وذلك للاقلال من تلوث العينات قبل تحليلها ، كيفية اعداد العينات و نقلها الي المعمل و حفظها الي حين تحليلها ، استبيان المواد الحافظة اللازمة لكل عنصر او مكون من مكونات الماء و مدة وطريقة الحفظ في حالة تعذر اجراء التحاليل المطلوبة في حينها . وقد تم توثيق هذه الورقة بالعديد من الرسومات التوضيحية و الجداول و المراجع العلمية الحديثة .