VIII: Appendices

Appendix 1: Analysis

As you must suspect, this appendix is not about talking to psychiatrists. It is about finding out the various simple physical and chemical truths we might want to know when dealing with biogas. It stresses analyses which can be simply undertaken, since, for the most part, complex analysis requires extensive background information and expensive equipment. Further, information on complex analysis would be redundant, since it exists in sufficiently clear form elsewhere. So, this chapter will talk about analyses which can be undertaken with simple tools and inexpensive chemicals and equipment.

One indispensable piece of equipment is a scale, for weighing small (under a kilogram or two pounds) amounts. A postal scale will work.

i. Gas Analysis

Biogas consists of only two important kinds of gases most of the time and it is these with which we should be concerned. There are the flammable and the nonflammable gases. Coincidentally, the major constituents of almost any biogas – CH_4 and CO_2 – fall neatly into these two categories.

Since CH₄ and CO₂ generally comprise 98% or 99% of most biogas, we can get a reasonable estimate of the amount of CH₄ in biogas by simply extracting the CO₂, and assuming everything we have left is methane. Now, you may react negatively to the idea of determining how much CH₄ you have by assuming that everything that isn't CO₂ is CH₄. "What about H₂S, H₂O, N₂, and NH₃?" you may feel compelled to cry out.

Here's the straight information: at this level of science, care and awareness are much more important than equipment. We can only expect a certain level of accuracy. With much better equipment, we might be able to better that by 5%. To get still greater accuracy, we'd have to spend quite a bit more money on equipment, and be quite a bit more competent.

In any case, nothing else we measure (volume or the heat requirements for example) is any more accurate, so why worry? We could go through all kinds of gymnastics, figuring out the partial pressure of water vapor and the amount of CH₄ which will dissolve in the solution we are using, but we won't. For those who wish to, some information is presented in the charts and tables which follow, but the rest of us will be happy with the simple, good-enough-for-government-work approach outlined below.

ii. CO₂, CH₄

First, fix up a jar or bottle so that it has a definite volume. Any glass jar will do as long as it has a lid that fits well and seals tightly. It can be calibrated for volume by weighing it partly filled with water, and then weighing it while empty. The volume of water which was in it can then be calculated by using the formula that follows.

$$V_j = \frac{Mt - Mj}{Dw}$$

where:

Vj = volume of the jar (to whatever point it's filled with water)

Dw = density of water (one kilogram per liter, 8.3 pounds per gallon)

Mt = mass (or weight) of water and jar

Mj = mass (or weight) of the dry empty jar

Subsequent calculations will be easier if you aim for a specific volume, such as a liter, or a quart, rather than 0.658 gallons, or some other such absurdity. Obviously therefore, it may be beneficial to start with a jar which already rates as being approximately some particular unit volume, or indeed which is already marked according to its volume.

After you determine the right volume of water, mark the jar in 3 places (around its circumference) at the bottom of what is called the meniscus. Use a grease pencil, or some other kind of marker that will not wash off the glass. Shown in Fig. A1.1 is a very narrow jar. In narrow glass jars, the meniscus is more visible than in wide jars.

Then fill the jar with clear cold water, invert it into a larger vessel filled with water, and bubble the biogas up into it to the particular measured volume you calibrated. (Remember that the bottom of the meniscus should just hit the line, like before.)

Now comes the tricky part: put the lid on the jar and transfer its contents to another large container. This container should be partly filled with an alkaline solution – sodium hydroxide (NaOH) or calcium hydroxide (CaOH) and water. *Be careful.* Such solutions can avidly eat through your flesh, and a small splash can result in a myriad of holes in your clothes. Wear rubber gloves, *eye shields*, and a piece of plastic sheet for an apron; move slowly and have a large bucket full of vinegar and water ready to wash off any drops of the alkali that get on you. Make sure your gloves don't leak.

The solution in the container should be shallow enough so you can set the inverted jar on the bottom of the container and still have enough room to comfortably grab the bottom of the jar, and pull it out. Use a plastic bucket, if possible. Plastic generally won't react with the alkaline solution.

Have tongs ready to remove the lid or cap as soon as you get it under the surface of the solution. Practice this a few times with air in the jar and water in



Fig. A1.2 Gas Measuring Apparatus



Fig. A1.1 Meniscus (cutaway view)

the larger container until you're sure you can do it safely and with skill. When going through the calibration process, swirl the jar gently to mix the water in the jar with the solution in the large container, being careful not to get air into the jar, or alkali solution out into the world at large.

Then just leave it alone. Come back occasionally, put on your protective equipment, slow yourself down, and gently swirl the jar a bit more. After a day or so, come back (put on your equipment) and lift the jar a bit, being careful not to get air into it. Notice that the biogas has less volume. This is because the CO₂ (hopefully, all of it) has reacted with the solution and has been pulled out of the biogas.

Using your tongs, find the lid on the bottom of the large container, set the jar in it, and twist the jar to put it back on, tightly enough so it won't fall off. Take some care with this step so you feel confident the lid will not fall off when you take the jar out of its container.

Bring over a container filled with water (don't take the jar over to it) and put the jar in it. Tighten the lid. Carefully wash it off.

Still using the rubber gloves, lift the jar out and set it, bottom down, lid up, on a level surface. Mark the bottom of the meniscus in three places, as before. With all the respect you have developed for the alkali

solution, gently empty the jar's contents into the alkali container, and again wash the jar in the water container. You no longer need to be concerned with the gas in the jar, as you already have the marks you need. Don't wash off the marks.



Fig. A1.3: H₂S Detection

Fill the jar with water up to the new marks, weigh it, and using the formula for Vj that appeared earlier in the chapter, calculate the volume of gas left in the jar. The approximate percentage of CO_2 is:

 $\% \text{CO}_2 = \frac{(\text{Vb} - \text{Ve})100}{\text{Vb}}$

where:

Vb = beginning gas volume

Ve = ending or final gas volume

And, of course, the assumption is that:

100 - %CO₂ = % CH₄

See Chapter 29: Gas Requirements, p. 127, for a means of making this information useful.

iii. H₂S

It just so happens that there is an inexpensive, moderately accurate method of determining the percentage of H₂S in biogas. It was developed by McBride and

Edwards and reported by Hazeltine (1933). Unfortunately, the test as developed will only tell if H₂S is present in amounts ranging from a trace to 15% of the maximum amount in which we are interested. In other words, the upper limit of sensitivity of the test as described and developed is 0.015% H₂S by volume, rather than the critical concentration of 0.100% H₂S by volume which we have taken as the upper limit of safe use in an engine. (See page 116 for further information.)

The basis of the test is that lead acetate – $Pb(COOH)_2$ – reacts with H₂S to form brown-colored lead and sulfur compounds. Thus, 22 by 77 millimeter (seven-eighths of an inch by three inches) strips of paper, soaked in a 10% solution of lead acetate (e.g., 10% lead acetate, 90% water by

Color	Percentage H2S
No color	0.0004 or less
Trace of color	0.0005 to 0.0008
Light color	0.0015 to 0.0025
Moderate color	0.005 to 0.008
Dark color	0.015 or more
Table Al-1 Estimation of Percentage of H ₂ S	

weight), dried, and then exposed to one liter of biogas, using the apparatus shown, would develop a characteristic brown coloration, as follows in Table A1.1.

It seems clear that, since these colors are the result of a certain amount of H₂S reacting with lead acetate, he test could be modified slightly so that if a dark brown were present, the test could be run again with *half* the former volume of biogas. Twice the above percentages of H₂S would then produce the color densities listed; this would give us a better idea about whether scrubbing was indicated or not. The test can also be used to indicate the effectiveness of scrubbing.

iv. Substrate Analysis

Most of us will not be able to do very much substrate analysis, as most substrate analysis requires rather elaborate equipment and technical knowledge.

Percentage of moisture

However, one quite important analysis which is also very simple is the percentage of moisture, or dry weight. All it requires is to weigh some amount of ordinary (moist or wet) substrate, then dry it in an ordinary oven (at around 220°F, or 105°C) for several hours. The dried weight is, of course, TS, total solids. My suggestion, if the substrate is as nasty as some are – pig manure for example – is *not* to use your kitchen oven. But hey, you might not be married, eh? (On the other hand, if you were happily wed before you dried the pig manure in the kitchen oven, the odds of still being married after are low...)

To figure the percentage of water, simply divide the weight of water by the total weight of the wet substrate, and multiply by 100:

$$\%H_2O = \frac{Ww - Wd}{Ww} \ge 100$$

where:

Ww = weight of wet substrate

Wd = dry weight of substrate

In Chapter 9: C/N, p. 35, we were working with what we called the "H₂O number," designated here as H*n* and simply the reciprocal of the percentage of H₂O, times 100:

$$H_2On = \frac{100}{\%H_2O}$$

If you're starting with wet and dry weights, an easier way to directly derive the H*n* is to divide the wet weight of the substrate by the weight of water:

$$Hn = \frac{Ww}{Ww - Wd}$$

(Likewise, the "CN number," designated here as CN*n*, is the reciprocal of that percentage proportion of the substrate which is C+N times 100. See the C/N chart on page 37.)