MEASUREMENT OF MOISTURE CONTENT IN SEEDS OF SOME NORTH AMERICAN HARDWOODS

F. T. Bonner

Forest Tree Seed Laboratory, Southern Forest Experiment Station,
USDA Forest Service, State College, Mississippi, U.S.A.

Preprint 51

Introduction

Current International rules (International Seed Testing Association, 1966) for determination of moisture content specify the air-oven method at 105 °C for all tree seeds except those of *Abies*, *Cedrus*, *Fagus*, *Picea*, and *Tsuga*, for which the toluene distillation method must be used. Calibration of air-oven methods against a good reference method, such as toluene distillation, has frequently been recommended, but data have been published for only a few species (Buszewicz, 1962; Hart and Golumbic, 1966). Seeds of major North American hardwoods are being studied at the Forest Tree Seed Laboratory at State College, Mississippi, and this paper summarises data on moisture-content measurement for three important species: *Fraxinus pennsylvanica* Marsh, *Liquidambar styraciflua* L., and *Platanus occidentalis* L. The studies had three objectives: to determine suitable air-oven schedules by comparison of air-oven measurements with toluene measurements on the same seed lots; to test the suitability of an electronic moisture meter with these species; to establish equilibrium moisture contents for seeds stored at various humidities and temperatures.

Materials and methods

General. The seeds were all from local collections in 1969. Within 6 months after collection, each lot was thoroughly mixed and divided into 25 sublots. Half of the sublots were dried to varying degrees by exposure for up to 48 hours in an air-conditioned room. The other half were placed in a humid cabinet at room temperature to absorb extra moisture; varying the times in the cabinet gave varying moisture levels. All sublots were then put into polyethylene bags for about 20 days of equilibration at 3 °C. *Selection of air-oven schedules.* The procedures for choosing air-oven schedules were largely those devised by Hart and Golumbic (1966) for a similar study. Sufficient samples were drawn from four of the sublots of each species to allow measurements of moisture content after 1, 2, 4, 6, 8, 12 and 24 hours of drying at 80°, 105°, or 130°C. Samples were weighed to the nearest 0.1 mg in tared bottles and dried in shallow aluminium dishes. Drying was measured from the time the ovens returned to their set temperatures after the doors were opened; delay was normally 10 to 15 minutes. At the specified times, and before reweighing, the dried samples were transferred for cooling to desiccators containing anhydrous $CaSO_4$. The cooling period was 2 hours. Shorter periods were tried, but cooling was apparently incomplete and the samples absorbed moisture from the air.

Loss in weight over time was calculated as a percent of fresh weight, and the four-lot average loss at each temperature was plotted against drying time.

Moisture contents of the same four lots were determined by toluene distillation on duplicate samples of about 20 g each. The methods of the Association of Official Agricultural Chemists (1965) were essentially followed. Distillation was for approximately 4 hours into a 5.0 ml Dean and Stark receiver. Moisture was read to the nearest 0.05 ml. Blanks with 2.0 ml of water were run to check the procedure; recovery was always 1.95 to 2.00 ml on the blanks. A horizontal line corresponding to the mean moisture content as determined by toluene distillation was added to the time-temperature curves (fig. 1). Points of intersection of this line with the time-temperature curves were taken as air-oven schedules for additional testing.

Moisture contents of the remaining 21 lots were then determined by toluene distillation and by the chosen air-oven procedure. Chi-square tests were used to indicate the accuracy of the air-oven procedures to within 0.5 or 1.0 percentage point of the true values (Freese, 1960).

Suitability of the electronic meter. An electronic meter manufactured by Motomco, Inc., Clark, New Jersey, U.S.A., was selected for this portion of the study.[1] This meter is used by the Grain Division, Agricultural Marketing Service, U.S. Department of Agriculture. Although built for use with grains, it has proved effective with seeds of several tree species (Hart and Golumbic, 1966).

Meter readings were taken on sublots of *L. styraciflua* and *P. occidentalis* and compared with determinations by the air-oven methods selected previously. The

[1]) The mention of firm names or products is solely for information, and does not imply endorsement or recommendation by the U.S. Department of Agriculture over other firms or similar products not mentioned.

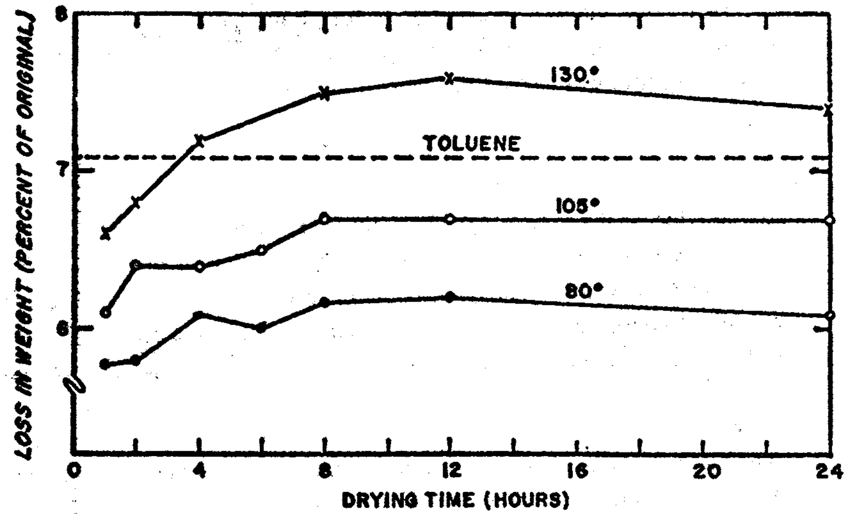

FIG. 1. Time-temperature curve for *P. occidentalis*. Each point represents the average of four samples. Toluene value was 7.1

regression of moisture content on meter reading was calculated and curves were constructed.

Because of the size and shape of the samaras, the meter could not be used with *F. pennsylvancia*.

Equilibrium moisture contents. To determine equilibrium moisture contents, samples of 1 to 2 g were taken in triplicate from a sublot of median moisture level for each species and placed in desiccators over saturated salt solutions at two temperatures. Relative humidities for the several salt solutions and temperatures were:

Salt		5°C	25°C
		RH%	
$CaCl_2$	$\cdot\ 6H_2O$	39.8	–
CrO_3		–	39.2
NaBr		–	57.0
NaCl		–	75.8
$BaCl_2$	$\cdot\ 2H_2O$	–	91.2
$ZnSO_4$	$\cdot\ 7H_2O$	94.7	–

The seeds were allowed to equilibrate for about 14 days, and then moisture contents were determined by the air-oven method selected previously. The entire procedure was carried out twice.

RESULTS

Examination of the time-temperature curves resulted in a choice of 4 hours at 130° as the air-oven method for the three species. Figure 1 shows the curves for *P. occidentalis*, which were typical for all three. Other drying schedules, for example, 15 hours at 105° for *F. pennsylvanica*, could have been chosen for any of the species, but a common time and temperature were picked for the sake of uniformity.

Mean values for air-oven vs. toluene measurements were very close (table 1). Chi-square analyses (0.05 level of significance) with toluene distillation data taken as true moisture contents indicated that 4 hours of drying at 130° was accurate to within 0.5 percentage point for *P. occidentalis* and to within 1.0 percentage point for the other two species. Overnight drying (15 hours) at 105° was accurate to within 1.0 percentage point for *F. pennsylvanica*.

TABLE 1. Summary of air-oven vs. toluene measurements. Air-oven method: 4 hours at 130°, 2 hours' cooling

Species	Samples No.	Range of sublot moisture contents by toluene method (%)	Mean moisture content (%)		Mean deviation of air-oven from toluene (%)
			Toluene	Air-oven	
F. pennsylvanica	25	8.33–17.92	10.42	10.41	0.09
L. styraciflua	25	6.40–19.70	10.91	10.48	−.43
P. occidentalis	24	6.13–17.29	9.89	9.92	.02

Because of the size of the *F. pennsylvanica* samaras, a comparison of intact with cut-up fruits was made with values from toluene distillation. The cutting was done very quickly with scissors immediately after initial weighing. Even then, duplicate measurements on nine sublots (table 2) showed that some moisture was lost during cutting. Only intact samaras should be used for this species.

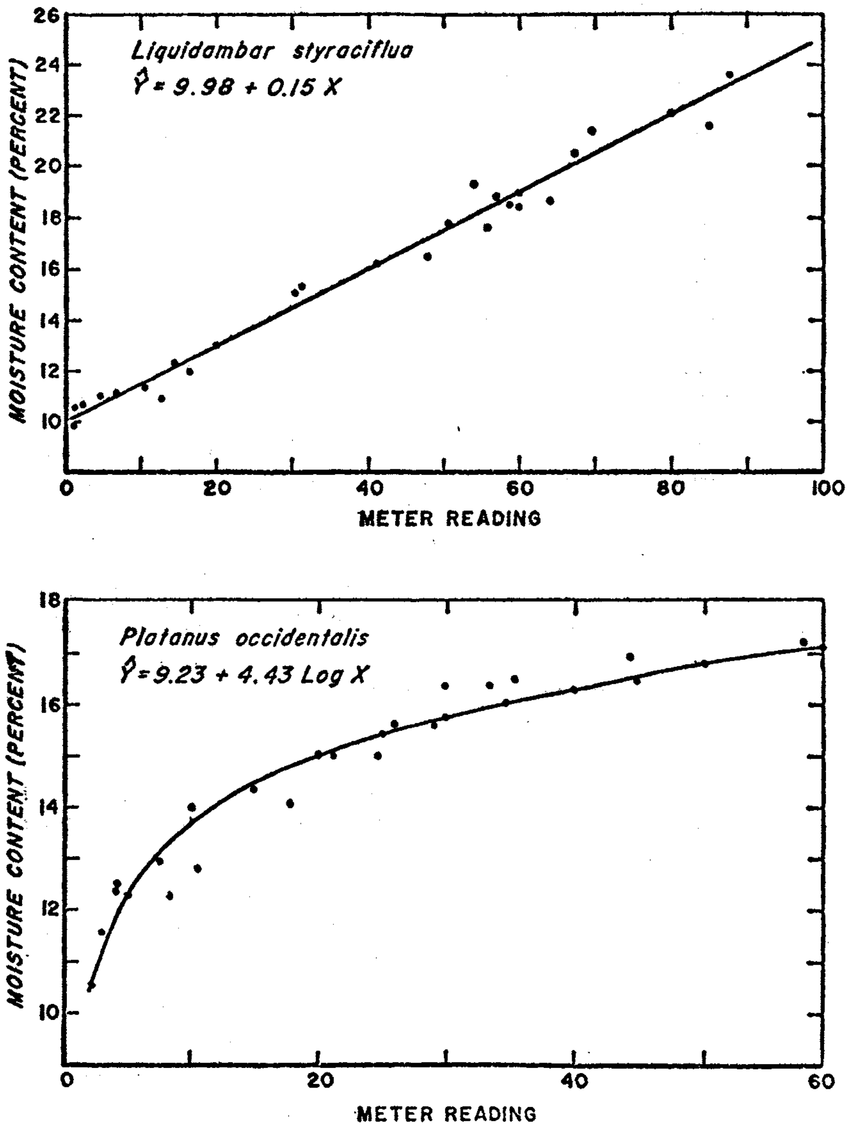

FIG. 2. Relationship of seed moisture content (in percent of fresh weight) to electronic meter reading. Y = moisture content and X = meter reading.

TABLE 2. Percentage moisture contents of intact and cut-up F. pennsylvanica samaras, as determined by toluene distillation

Samaras cut	Samaras intact
7.84	8.96
8.25	8.33
7.84	7.77
10.78	12.02
10.89	11.32
10.78	12.74
10.89	12.62
10.44	12.28
11.88	15.53

Suitability of the meter. For *L. styraciflua* the relationship of moisture content to meter readings was linear (fig. 2). With 100 g samples, moisture contents as low as 10% could be measured (table 3).

For *P. occidentalis* the regression was curvilinear. A logarithmic relationship seemed appropriate (fig. 2). As with *L. styraciflua*, maximum loading of the sample chamber was necessary to obtain readings at lower moisture contents, and even then the total measurable range was only 11.6 to 17.2% (table 3). Despite these limitations, the meter can be useful with *L. styraciflua* and *P. occidentalis.*

TABLE 3. Relationship between moisture content and Motomco meter readings for *L. styraciflua* and *P. occidentalis*. In regressions, Y = moisture content and X = meter reading

Species	Sample No.	Sample weight g	Range of measured moisture content %	Regression equation	r^2	Standard error of estimate %
L. styraciflua	24	100	9.8–23.6	$Y = 9.98 + 0.15\,X$	0.9767	±0.66
P. occidentalis	18	90	11.6–17.2	$Y = 9.23 + 4.43 \log X$	0.9220	±0.54

Equilibrium moisture contents. Moisture contents of seed equilibrated under the several temperature and humidity levels are summarised in Table 4. At any one level the values are very close for all three species. The table probably indicates moisture levels that seeds will reach if stored in open containers under the conditions specified.

TABLE 4. Equilibrium moisture contents at 5° and 25°C, and various humidities

Temperature (C.)	Relative humidity (%)	Seed moisture content (%)		
		P. occidentalis	*L. styraciflua*	*F. pennsylvanica*
25	39	8.0	7.1	7.7
25	57	10.5	8.5	9.1
25	76	12.8	11.1	12.6
25	91	19.3	15.9	20.1
5	40	9.2	8.3	8.3
5	95	20.8	19.5	23.0

DISCUSSION

Measurement by air-oven methods to within 0.5 percentage point of toluene measurements was the accuracy goal of this study, and was achieved with *P. occidentalis*. For *L. styraciflua* and *F. pennsylvanica*, accuracy was within 1.0 percentage point, which is good enough for seed-processing and storage decisions. Where greater accuracy may be desirable, as in some research, a laboratory technique must be employed. In addition to toluene distillation, the Karl Fischer method as modified by Hart and Neustadt (1957) and near-infrared spectrophotometry of methanol extracts (Hart, Norris and Golumbic, 1962) have been described as good reference methods.

Readout on the electronic meter scale is a function of total moisture in the sample chamber, and the relationship of scale reading to moisture varies according to the amount of material in the chamber. Calibration curves must be established with samples of a constant weight. The larger the sample weight, the lower the moisture percentage that can be determined from the scale reading. For large seeds, an increase in size of the sample chamber would be desirable to extend the measurement range.

Hart and Golumbic (1966) found a slight temperature effect on performance of the meter. In a modern heated and air-conditioned building, the temperature effect can probably be ignored, as long as the seeds have been allowed to come to instrument (room) temperature.

The equilibrium moisture contents listed in Table 4 are similar to values published for several conifers (Bartels, 1956; Barton, 1941) and some crop seeds (Welch, 1967). When RH was about equal, differences between 5° and 25°C were not great. Welch (1967) stated that a drop of 11°C in temperature will increase seed moisture approximately 2%.

Summary

An air-oven schedule of 4 hours at 130°C, followed by 2 hours of cooling in a desiccator, gave moisture contents within 0.5 percentage point of the true value for *Platanus occidentalis* and within 1.0 percentage point for *Liquidambar styraciflua* and *Fraxinus pennsylvanica*. This degree of accuracy should be sufficient for processing and storage decisions. An electronic meter was effective for *Platanus occidentalis* seeds in the range from 12 to 17% and for *Liquidambar styraciflua* in the range from 10 to 24%. Equilibrium moisture contents at several temperatures and humidities were very close to published values for seed of conifers and agronomic plants.

Résumé

Mesure de la teneur en eau des semences de quelques espèces d'arbres feuillus d'Amérique du Nord

Un séchage à 130°C pendant 4 heures dans une étuve, suivi d'un refroidissement de 2 heures dans un déssiccateur a fourni des pourcentages de teneur en eau différant de la vraie valeur de moins de 0.5 pour *Platanus occidentalis* et de moins de 1.0 pour *Liquidambar styraciflua* et *Fraxinus pennsylvanica*. Ce degré de précision devrait être suffisant pour prendre des décisions en matière de conditionnement et de stockage. Un appareil de mesure électronique s'est avéré efficace pour les semences de *Platanus occidentalis* dans la gamme de 12 à 17% de teneur en eau et pour *Liquidambar styraciflua* dans la gamme de 10 à 24%. Les teneurs en eau à l'équilibre, pour des températures et des dégrés hygrométriques différents, ont été en accord très étroit avec les valeurs publiées pour les semences de conifères et d'espèces agricoles.

Zusammenfassung

Messungen des Feuchtigkeitsgehalts von Samen einiger nordamerikanischer Hartholzarten

Eine Trockenschrankmethode von 4 Stunden bei 130°C, gefolgt von 2 Stunden Kühlung in einem Exsikkator, lieferte Feuchtigkeitsgehalte für *Platanus occidentalis* innerhalb der 0.5-Grenze des wahren Wertes, und für *Liquidambar styraciflua* und *Fraxinus pennsylvanica* innerhalb der 0.1-Grenze des wahren Wertes. Dieser Genauigkeitsgrad sollte für Aufbereitung und Lagerungsentscheidungen ausreichen. Ein elektronisches Meßgerät erwies sich für *Platanus occidentalis*-Samen im Bereich von 12–17% und für *Liquidambar styraciflua* im Bereich von 10–24% als aussagefähig. Wassergehalte, welche bei mehreren Temperatur- und Feuchtigkeitsstufen im Gleichgewicht waren, legen sehr nahe an den für das Saatgut von Nadelhölzern und von landwirtschaftlichen Pflanzen veröffentlichten Werten.

References

Association of Official Agricultural Chemists. (1965): *Official methods of analysis.* Ed. 10, (Ed. W. Horwitz) A.O.A.C., Washington, D.C.

Bartels, H. (1956): Über den Wassergehalt der Nadelholzsamen. *Forstwiss. Centralbl.* 75, 124–128.

Barton, L. V. (1941): Relation of certain air temperatures and humidities to viability of seeds. *Contr. Boyce Thompson. Inst. Pl. Res.*, 12, 85–102.

Buszewicz, G. (1962): A comparison of methods of moisture determination for forest tree seeds. *Proc. Int. Seed Test. Ass.*, 27, 952–970.

Freese, F. (1960): Testing accuracy. *Forest Sci.*, 6, 139–145.

Hart, J. R. and Golumbic, C. (1966): The use of electronic moisture meters for determining the moisture content of seeds. *Proc. Int. Seed Test. Ass.*, 31, 201–212.

Hart, J. R. and Neustadt, M. H. (1957): Application of the Karl Fischer method to grain moisture determination. *Cereal Chem.*, 34, 26–37.

Hart, J. R., Norris, K. H. and Golumbic, C. (1962): Determination of the moisture content of seeds by near-infrared spectrophotometry of their methanol extracts. *Cereal Chem.*, 39, 94–99.

International Seed Testing Association (1966): International rules for seed testing. *Proc. Int. Seed Test. Ass.*, 31, 1–152.

Welch, G. B. (1967): Seed storage to maintain quality. *Short course for seedsmen.* 1967, 53–63. Miss. State Univ.