

## LOUVAIN NATURAL RADIOCARBON MEASUREMENTS I\*

J. M. DOSSIN, J. M. DEUMER, P. C. CAPRON

Department of Nuclear Chemistry, University of Louvain, Louvain, Belgium

The following list covers the samples measured at the Louvain  $C^{14}$  dating laboratory during a period from May to December 1961.

### INTRODUCTION

*Pretreatment of samples*—Foreign material (rootlets and other contaminating matter) is removed before chemical treatment is started. Samples are boiled with dilute HCl (1%) for one hour, washed with distilled water, boiled with dilute NaOH (1%) overnight, washed again with distilled water, reboiled with dilute HCl (1%) for one hour and dried in an oven. This treatment should remove both carbonate and humic contamination (Barendsen, 1955; de Vries, 1958).

*Preparation of filling gas*—Samples are burned and the  $CO_2$  evolved is purified according to the standard procedures (de Vries, 1956; Münnich, 1957; Van Pee, Crèvecoeur, and Capron, 1958).  $CO_2$  is converted to methane in one step by catalytic hydrogenation using a nickel catalyst (Coppens, 1934; Nicolaï, D'Hont, and Jungers, 1946; Diethorn, 1956; Dossin, and Deumer, 1961). After thorough purification,  $CH_4$  is kept in store to check radon contamination.

*Counter design*—The sample is introduced to the counter as purified  $CH_4$ , at a pressure of 3 atm. The counter, of stainless steel, has a sensitive volume of 0.6 L. It is shielded with 40 cm of iron, 2.5 cm of lead and an annular G.M. counter with 13 anodes, coupled in anticoincidence with the proportional counter (Raeth, Sevold, and Pederson, 1951; de Vries, 1956; Berdau, 1960).

*Experimental procedure*—The proportional counter plateaus are ca. 1800 v long and have a slope of less than 0.4% per 100 v. With this counting equipment, the background is 7.3 counts/min. As modern standard, Munaut provided tree rings from A.D. 1870 to A.D. 1890, taken from an oak tree that grew in Arenberg Park, Heverle. In this way errors due to atmospheric depletion (Suess, 1955) and to past changes in  $C^{14}$  concentration (de Vries, 1958; Münnich and Vogel, 1958) have been minimized. Activity of the modern standard is 17.8 counts/min. Counter efficiency is 71% (Anderson, and Libby, 1951).

*Calculation*—All samples are measured twice for at least 18 hr, two weeks apart. **The value 5760 yr has been used for the half life of  $C^{14}$**  (determined by NBS, 1961). The results are expressed in years before 1950; no  $C^{13}$  corrections are made. The  $\sigma$  errors given include the experimental standard deviation ( $\sigma_{\text{exp}}$ ) on the counting rate of the unknown sample, the modern

\* Because some Louvain dates have been published elsewhere, all are quoted in terms of the NBS half life of  $C^{14}$ , 5760 yr. Subsequent corrections, probably to be adopted in 1962, will probably be smaller than corrections of dates based on Libby's value, 5570 yr, but will almost certainly be necessary.

value and the background. When the activity is very low,  $4\sigma_{\text{exp}}$  has been used instead of  $\sigma_{\text{exp}}$  (Crèvecoeur, Vander Stricht, and Capron, 1959).

#### ACKNOWLEDGMENTS

The authors express their thanks to E. H. Crèvecoeur for helpful discussions (though he was on leave of absence during a part of the time of this work), and to J. M. Marlaire and G. Michotte for their assistance in preparing and measuring the samples.

Financial support was provided by the "Institut Interuniversitaire des Sciences Nucléaires."

#### SAMPLE DESCRIPTIONS

##### I. GEOLOGIC SAMPLES

##### Lommel series

Peat layer from 195 to 207 cm below surface of the plain of Weyerkenne Bergen ( $51^{\circ} 15' \text{ N Lat}$ ,  $5^{\circ} 18' \text{ E Long}$ ), near Lommel, Campine, Belgium. Overlies sand correlated with the Würm glaciation and overlain by frost-contorted sand. Pollen analysis by W. Mullenders and M. Cornil indicates a temperate climate between two cold periods. Coll. 1957 and subm. by F. Gullentops (Gullentops, Mullenders, Dossin, Crèvecoeur, Deumer, 1962).

**Lv-100. Lommel 32/2B**  **$11,640 \pm 250$**

Peat from 197 cm.

**Lv-101. Lommel 32/2B**  **$12,310 \pm 340$**

Peat from 206 cm.

**Lv-6. Lommel 32/2B**  **$10,380 \pm 420$**

Birch wood (*Betula*) id. by R. Antoine from peat at 205 cm. *Comment*: no pretreatment.

**Lv-56. Tontelange Faascht I**  **$1130 \pm 190$**

Peat from Tontelange ( $49^{\circ} 44' \text{ N Lat}$ ,  $5^{\circ} 48' \text{ E Long}$ ), Prov. of Luxembourg, Belgium, 80 to 85 cm below surface. Pollen analysis shows, at this horizon, an increase of 13% of Pine (*Pinus sylvestris*) and a beech maximum (*Fagus sylvatica*) of 25%. The age of the beech maximum ( $F_2$  Hautes Fagnes) identified by Dricot (1960) from pollen analysis, seems to agree with the date. Coll. 1958, and subm. by M. Coûteaux.

##### Beerse series

Peat from Algemeen Bouw Bedrijf quarry, near the Rijkervorsel-Beerse road ( $51^{\circ} 20' 15'' \text{ N Lat}$ ,  $4^{\circ} 48' 52'' \text{ E Long}$ ), Prov. of Anvers, Belgium. Section shows: 0-230 cm, Late Glacial dune sand, horizontally stratified, covered by a humic ferric podzol; 230-252.5 cm, compact peat with disturbance of Brödelboden type; below 252.5 cm Pleniglacial coversand of Würm age. Coll., pollen studied, and subm. 1961 by J. De Ploey.

**Lv-73. Beerse, 230 to 235 cm 10,920  $\pm$  540**

Peat from 230 to 235 cm. Pollen in this sample represents Alleröd zone. The date agrees with previous Alleröd dates (Gross, 1958).

**Lv-74. Beerse, 240 to 245 cm 11,950  $\pm$  430**

Peat from 240 to 245 cm. Pollen in this sample represents Older Dryas zone. The date agrees with previous Older Dryas dates (Gross, 1958).

**Lv-75N. Beerse, 247.5 to 252.5 cm 12,160  $\pm$  415**

Peat from 247.5 to 252.5 cm. Pollen in this sample represents Bölling zone. The date agrees with previous Bölling dates (Gross, 1958).

**Lv-75. Beerse, 247.5 to 252.5 cm 10,690  $\pm$  465**

Sample from same horizon as Lv-75N. *Comment*: no pretreatment; the check shows that the sample was contaminated by younger material, perhaps humic acids, and that this can be extracted by standard pretreatment.

## II. ARCHAEOLOGIC SAMPLES

**Lv-10. Via Mansuerisca 4070  $\pm$  160**

Peat from a position beneath the road called Via Mansuerisca which leads from Trêves to Maastricht, across the Hautes Fagnes (50° 31' N Lat, 6° 03' E Long), Belgium. Subm. by J. Mertens, Service des Fouilles de l'Etat, to help fix the very discussed age of this road (Bastin, 1935). Sample was taken from a horizon under the road rather than from a wooden sample which could have been used for a repair of the road. It looks as if this sample was not good because taken from a horizon too deep under the road. Another sample is clearly necessary for ascertaining the age.

**Lv-43. St Donatius Cathedral 870  $\pm$  140**

Wood from a tree trunk (beech) used in the basement of the pre-Romanesque construction of the St Donatius Cathedral, Bruges (51° 13' N Lat, 3° 13' E Long). Coll. 1955 and subm. by J. Mertens. *Comment*: no pretreatment. From historical considerations, dated at the end of 9th or at the beginning of 10th century.

**Leopoldville series**

Samples related to prehistoric industries in the Leopoldville plain. Coll. by H. V. Moorsel, Prehistorical Mus., Lovanium Univ., Leopoldville, Congo; subm. by F. Gullentops, Univ. of Louvain.

**Lv-44. Lemba >30,000**

Fossil wood from Lemba (4° 23' S Lat, 15° 20' E Long), Prov. of Leopoldville, Congo; in peaty sandstone, the surface of which has a late Lumpemban culture. Sandstone is covered by sand which has a Tshitolién culture.

**Lv-45. Mt. Gafula 6030  $\pm$  190**

Charcoal from Mt Gafula (4° 28' S Lat, 15° 13' E Long), Prov. of Leo-

poldville, Congo, in sand at depth 2 m. Sample is from a cutting factory characteristic of late Tshitoliën.

**Lv-46. Mt. Amba** **3080 ± 170**

Charcoal from Mt. Amba, Lovanium Plateau (4° 26' S Lat, 15° 18' E Long), Prov. of Leopoldville, Congo, from a sterile layer. The same layers of charcoal often occur around Leopoldville, in yellow sand, at depth 1 m. *Comment*: charcoal is probably a consequence of clearing of forest.

**Lv-47. Basoko River** **>30,000**

Charcoal from a site near Basoko River on Leopoldville plain (4° 21' S Lat, 15° 17' E Long), Prov. of Leopoldville, Congo. Sample is from a Lupembian horizon (intermediate zone) overlain by clayey silt and 30 cm above polymorphous sandstone blocks.

**Eprave series**

Charcoal from a burned horizon, remains of the Roman camp at Eprave (50° 08' N Lat, 4° 09' E Long), Prov. of Namur, Belgium. Coll. 1958; subm. by J. Mertens. Coins dating from the end of the 4th century were found at this horizon.

**Lv-54. Ep. 29** **1780 ± 100**

**Lv-55. Ep. 25** **<200**

*Comment*: C<sup>14</sup> date is much too young: the discrepancy is unexplained.

**Lv-93. Deir el Bahari** **2750 ± 210**

Wood from the lid of an Egyptian mummiform coffin, found at Deir el Bahari, W of Thebes (Luxor) Egypt. Although the coffin dates from the end of the 22nd Dynasty, the style of the lid suggests a date between 1000 and 750 B.C. (Custers), in agreement with the C<sup>14</sup> date. Coll. by Musée Biblique, Inst. d'Archéologie, Univ. of Louvain; subm. by J. Mertens. *Comment*: sample not pretreated.

REFERENCES

- Anderson, E. C., and Libby, W. F., 1951, World-wide distribution of natural radiocarbon: Phys. Rev., v. 81, p. 64-69.
- Bastin, J. (abbé), 1935, La Via Mansuerisca: Spa Editions "J'ose," 15 p.
- Barendsen, G. W., 1955, Ouderdomsbepaling met radioactieve koolstof: Thesis, Univ. Groningen, September 20, 1955, 88 p.
- Berdau, D., 1960, Entwicklung einer hochempfindlichen Antikoinzidenz-Zählapparatur für sehr schwache  $\beta$ . Strahlung: Heidelberg, Ingenieurarbeit.
- Coppens, L., 1934, Contribution à l'étude des gaz naturels et en particulier des grisous: Soc. Chim. Belgique Bull., v. 43, p. 335-360.
- Crèvecoeur, E. H., Vander Stricht A., and Capron, P. C., 1959, Precision of the dating method. Standardization of the calculation of the errors and the maximum age in the C<sup>14</sup> method: Acad. royale Belgique Bull. cl. sci., v. 45, p. 876-890.
- De Ploey, J., 1961, Morfologie en kwartair-stratigraphie van de Antwerpse Noorderkempen: Ph.D. Thesis, to be pub. in Acta Geog. Lovaniensia [French abs.].
- Diethorn, Ward, 1956, A methane proportional counter system for natural radiocarbon measurements: Carnegie Inst. Technology, U. S. Atomic Energy Comm. Rept. NYO-6628, xi, 146 p. [processed].
- Dossin, J. M., and Deumer, J. M., 1961, Notes sur la détermination des âges par le radiocarbène: Univ. of Louvain, 106 p.

- Dricot, E. M., 1960, Recherches palynologiques sur le plateau des Hautes Fagnes: Soc. Royale de Bot. Belgique Bull., v. 92, p. 157-196.
- Gross, Hugo, 1958, Die bisherigen Ergebnisse von  $C^{14}$  Messungen und paläontologischen Untersuchungen für die Gliederung und Chronologie des Jungpleistozäns in Mitteleuropa und den Nachbargebieten: *Eiszeit. u. Gegenwart*, v. 9, p. 155-188.
- Gullentops, F., Mullenders, W., Dossin, J. M., Crèvecoeur, E. H., Deumer, J. M., 1962, Le tardiglaciaire de Lommel: Acad. royale Belgique Bull. cl. sci. [in press].
- Münnich, K. O., 1957, Messung Natürlich Radiokohlenstoffs mit einem  $CO_2$ -Proportional-Zählrohr. Einige Anwendungen der Methode: Ph.D. dissertation, Univ. Heidelberg [processed].
- Münnich, K. O., and Vogel, J. C., 1958, Durch Atomexplosionen erzeugter Radiokohlenstoff in der Atmosphäre: *Naturwissenschaften*, v. 45, p. 327-329.
- Nicolai, J., D'Hont, M., and Jungers, J. C., 1946, La synthèse du Méthane à partir d'anhydride carbonique et d'hydrogène sur le nickel: Soc. Chim. Belgique Bull., v. 55, p. 160-176.
- Raeth, C. H., Sebold, B. J., and Pederson, C. N., 1951, A multiple-anode anticoincidence ring counter: *Rev. Sci., Instruments*, v. 22, p. 461-463.
- Suess, H. E., 1955, Radiocarbon concentration in modern wood: *Science*, v. 122, p. 415-417.
- Van Pee, W., Crèvecoeur, E. H., and Capron, P. C., 1958, Détermination des âges par le radiocarbène: Acad. royale Belgique Bull. cl. sci., ser. 5e, v. 46,, p. 994-1007.
- Vries, Hessel de, 1956, Purification of  $CO_2$  for use in a proportional counter for  $C^{14}$  age measurements: *Appl. Sci. Research, sec. B.*, v. 5, p. 387-400.
- 1956, The contribution of neutrons to the background of counters used for  $C^{14}$  age measurements: *Nuclear Physics*, v. 1, p. 477-479.
- 1958, Radiocarbon dates for upper Eem and Würm-interstadial samples: *Eiszeit. u. Gegenwart*, v. 9, p. 10-17.
- 1958, Atomic bomb effect: Variation of radiocarbon in plants, shells, and snails in the past 4 years: *Science*, v. 128, p. 250-251.