# The Köln University Radiocarbon Laboratory (1963-2000)

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**Abstract** – In the course of the last 40 years the radiocarbon dating laboratory at the University of Köln has produced ca. 5,000 radiocarbon dates on archaeological samples, mainly of charcoal, wood and bone collagen from the European Bronze Age, Neolithic, and Palaeolithic periods. This paper presents a brief history of the laboratory in the years 1963-2000 and describes recent developments in dating equipment and technical procedures.

**Keywords** – Radiocarbon, History, Proportional Counter.

**Zusammenfassung** – Während der letzten 40 Jahre hat das Kölner $^{14}$C-Labor ca. 5.000 Radiokarbondaten an archäologischen Proben gemessen. Es handelt sich überwiegend um Holzkohle-, Holz- und Knochenproben aus der europäischen Bronzezeit, dem Neolithikum und dem Paläolithikum. Dieser Beitrag gibt einen kurzen Überblick über die Geschichte des Kölner Labors von 1963 bis 2000 und beschreibt die neuesten Entwicklungen bei den meßtechnischen Verfahren.

**Schlüsselwörter** – Radiokohlenstoff, Geschichte, Proportionalzählrohr.

## History

Once the occurrence in nature of a long-lived radioactive isotope of carbon $^{14}$C (half-life 5730 ± 30 a) had been recognized (ANDERSON et al. 1947), it did not take long until the first successful datings were demonstrated (ARNOLD & LIBBY 1949). All over the world, a large number of physicists, geologists, paleobotanists and prehistorians became interested in the new dating method.

At Köln, plans to build a radiocarbon dating laboratory where initiated in 1957 by Hermann Schwabedissen, following a prehistoric conference held at Münster (SCHWABEDISSEN & FREUNDLICH 1966). The equipment was designed and built (as well as financed) in the years 1957 to 1959 by K. Beckhoff, an electronic engineer and owner of an electroengineering plant. This initial equipment was transferred to the Universität zu Köln in December 1959. The Beckhoff equipment was soon working dependably, and a second counting system of similar design was built 1960-1962.

In January 1963 Jürgen Freundlich assumed responsibility for the laboratory. He was able to undertake routine datings by the middle of the year. The first Köln date list (SCHWABEDISSEN & FREUNDLICH 1966) was published three years later. It contained only 17 radiocarbon dates, but which were obviously well-selected. Up to this time actually 50 dates had already been measured and this short-cutting requires an explanation. Schwabedissen and Freundlich themselves (1966, 239) write that they only included such dates in the list that could be checked either by other laboratories or against independent (e.g. historic) age determinations. Looking back, we can guess on a further reason for the extreme caution in this early publication. That is the raging controversy between Hermann Schwabedissen and Vladimir Milojčić about the absolute dates of the European Neolithic (SCHWABEDISSEN & MÜNNICH 1958; MILOJČIĆ 1961).

Jürgen Freundlich ran the laboratory for altogether nearly 30 years (up to 1992) and under his charge the laboratory produced many thousands of archaeological radiocarbon dates, on which some quite large parts of the European neolithic chronology are based (BREUNIG 1983). One of the most important techniques he introduced was a change of the $CO_2$ purification method (FREUNDLICH & RUTLOH 1972), Initially, a quite efficient but rather tedious 'wet' $CO_2$ purification method had been used. As an alternative, the sample $CO_2$ can be efficiently purified by circulation over hot copper, according to a method originally devised in Groningen by Hessel de Vries (cf. VOGEL & WATERBOLK 1967). The 'De

Vries' copper method is used in the Köln laboratory still today.

A second major breakthrough in laboratory techniques came in the year 1970, with the installation of two additional proportional counters with previously unachieved low background values. These counters (called Zählrohr ZIII and ZIV) have been in operation ever since.

## Dating Procedures

Although the dating procedures (SCHWABEDISSEN & FREUNDLICH 1966; FREUNDLICH et al. 1980) of the Köln Radiocarbon laboratory have remained largely unchanged since 1970, in recent years there have been some noteworthy modifications in active shielding and electronics which are described in the following. To be complete, we first repeat the basic dating procedures. As mentioned, the laboratory is equipped with two proportional counters (ZIII and ZIV) which use purified $CO_2$ as a working gas. The counters are installed some 5 m below ground level in the sub-basement of the Institute of Prehistory in Köln. To further reduce the penetrating cosmic radiation, the counters are heavily shielded by 30/40 cm lead (sides/top) and 20 cm boron loaded paraffin. Working voltages of the counters are 7 000 Volt (ZIII) and 5 100 Volt (ZIV). The plateaus have length 500 Volt and slope $\leq$ 1%/100 Volt for $^{14}C$ beta radiation. The counters have active volumes of 0.7 liter and are filled to 3 atm with purified carbon dioxide.

Within the passive lead shielding, both counters have essentially the same meson count rate of ca. 120 cpm, with slight variations depending on barometric pressure. The counters are surrounded by 2 cm old lead, originating from a medieval lead window frame of Köln Cathedral. To be precise, this lead shielding is the only remaining part of a medieval window sadly destroyed during World War II. The counters are installed inside a large anticoincidence cosmic ray guard shield, with continuous slow flow of $Ar/CH_4$-(90/10 mixture). This guard counter was built by Hoffmann GmbH (Heidelberg) according to construction plans of the Heidelberg Radiocarbon Laboratory. It was installed in March 1998 to replace the earlier used shield of 32 commercial Geiger counters of the cosmic-ray type (model HZ-100, Zentralwerkstatt Göttingen). With cosmic guard switched on, and with the anticoincidence electronics set active, the counter backgrounds are reduced from 120 cpm to their nominal ($2\sigma$ = 95 % confidence) background values of 0.8 ± 0.02 cpm (ZIII) and 1.4 ± 0.02 cpm (ZIV). The system stability achieved with the new cosmic guard shield is most satisfactory, as demonstrated by the statistical behaviour of the background count rates measured over the last two years, the spread of which is close to Poisson (Tab. 1).

Also shown in table 1, counter backgrounds are regularly measured every 6–8 weeks with counting periods of 3 or 4 days, which corresponds to the typical 'Monday-Thursday-Monday' weekly rhythm used for unknown-age samples. For test and calibration purposes background counts are often extended, up to 2-3 weeks (e.g. 20 days in March 1998 after installation of the new cosmic guard). In rare cases background measurements are repeated at shorter intervals. An example is run Nr. 523 (27.-29.11.98) which was used to check on the unexpectedly low background previously measured in both counters during run Nr.521. Archaeological samples are typically measured twice, if possible with independent fillings using both counters, giving a measuring period of 6-8 days per sample. Extended counting periods are also applied to archaeological samples, judged to be unusually important, as shown in Fig.1 for an 8 day count (3093 ± 37 BP = first measurement) on wood charcoal (sample 34/78) from the Bronze Age site of Kastanas, submitted by Reinhard Jung (Berlin). When averaged with the second (3113 ± 44 BP) and third (3133 ± 25 BP) result, and corrected for $^{13}C$-fractionation, the resulting final date KN-5060: 3114 ± 19 BP has the precision we would like all radiocarbon dates on archaeological samples to have.

The dating precision typically achieved for samples with a 7 day count is 0.4 pmC (percent Modern Carbon), which translates to a standard deviation ± 32 BP (68-% confidence) for samples younger than 4000 yrs. Modern $^{14}C$ count rates (netto 0.95 Oxalic Acid) are 10.55 ± 0.01 cpm (ZIII) and 10.22 ± 0.01 cpm (ZIV). The system has dating limits of

| Nr. | Date | Run [days] | Counter ZIII [cpm] | Counter ZIV [cpm] |
|---|---|---|---|---|
| 467 | 25.03.98 | 20 | 0.815 ± 0.006 | 1.432 ± 0.008 |
| 468 | 14.04.98 | 2 | 0.796 ± 0.020 | 1.441 ± 0.028 |
| 474 | 04.05.98 | 7 | 0.811 ± 0.010 | 1.421 ± 0.013 |
| 489 | 27.07.98 | 4 | 0.796 ± 0.014 | 1.476 ± 0.019 |
| 506 | 25.09.98 | 3 | 0.810 ± 0.015 | 1.364 ± 0.020 |
| 521 | 20.11.98 | 3 | 0.737 ± 0.015 | 1.343 ± 0.020 |
| 523 | 27.11.98 | 3 | 0.771 ± 0.010 | 1.369 ± 0.013 |
| 536 | 25.01.99 | 3 | 0.758 ± 0.015 | 1.408 ± 0.020 |
| 558 | 26.04.99 | 4 | 0.790 ± 0.013 | 1.405 ± 0.108 |
| 559 | 30.04.99 | 3 | 0.797 ± 0.016 | 1.385 ± 0.021 |
| 577 | 12.08.99 | 4 | 0.769 ± 0.013 | 1.365 ± 0.018 |
| 579 | 19.08.99 | 4 | 0.757 ± 0.013 | 1.417 ± 0.018 |
| 594 | 18.10.99 | 4 | 0.792 ± 0.013 | 1.446 ± 0.018 |
| 601 | 15.11.99 | 3 | 0.766 ± 0.015 | 1.424 ± 0.021 |
| 621 | 31.01.00 | 3 | 0.768 ± 0.015 | 1.350 ± 0.020 |
| 645 | 04.05.00 | 4 | 0.786 ± 0.013 | 1.420 ± 0.018 |
| 660 | 17.07.00 | 24 | 0.7769 ± 0.005 | 1.4136 ± 0.007 |
| **Nominal Value (95% Confidence)** | | | **0.8 ± 0.02 cpm** | **1.4 ± 0.02 cpm** |

**Tab. 1** Background Measurements of Köln $CO_2$ Proportional Counters.
Period March 1998 to July 2000.

46 ka (counter ZIII) and 42 ka (counter ZIV). Dating capacity is ca. 120 dates per year.

## Chemical and Physical Sample Preparation

We routinely process samples from archaeological excavations, which are most often charcoal (minimum sample weight is 6 g), followed by bones (min 300 g) and wood (min 20 g). The sample pretreatment is typically as follows: after weighing, drying, and manual extraction of rootlets, charcoal and wood samples are washed in 5% HCl, then in 10% NaOH, followed again by 5% HCl. Between each of steps, the sample is rinsed to neutral with distilled water, and subsequently dried and weighed. For collagen extraction from bone we use a similar cleaning procedure, but with prolonged acid treatment and weaker HCL to decalcify the bone.

The counters use $CO_2$ as filling gas, which is extremely sensitive to electronegative gas impurities and must therefore be highly purified to ensure reproducible counting conditions. The sample is oxidised unter controlled conditions in $N_2/O_2$ and passed over hot copper oxide in the combustion tube. The resulting $CO_2$ is further sent through a wash bottle with saturated $KMnO_4$, dried in a high efficiency spiral columne at -78 °C, sent through an oven filled with hot silver wool, again dried at -78 °C, and stored overnight in a 6 L bulb. At this point the first 'De Vries' purification is applied.

To allow for alpha-decay of $^{222}Rn$ deriving from the U/Th-content of the sample, the sample gases are subsequently stored for at least 4 weeks. Prior to each counter filling, the sample is further purified in a second vacuum line, again using thermal circulation over hot 600 °C. Sample gases deriving from bone samples are given additional drying at -78 °C. Following this procedure the gases mostly (ca. 98%) have excellent counting properties.

Gas quality is routinely verified by measuring the electronic amplification using the meson flux at the half-value of the working voltage. In the rare cases (ca. 2 %), when a non-optimal gas purity would require a larger than 70 volt shift in working point, the sample gas is immediately taken out of the counter and further purified by recirculation over hot copper,

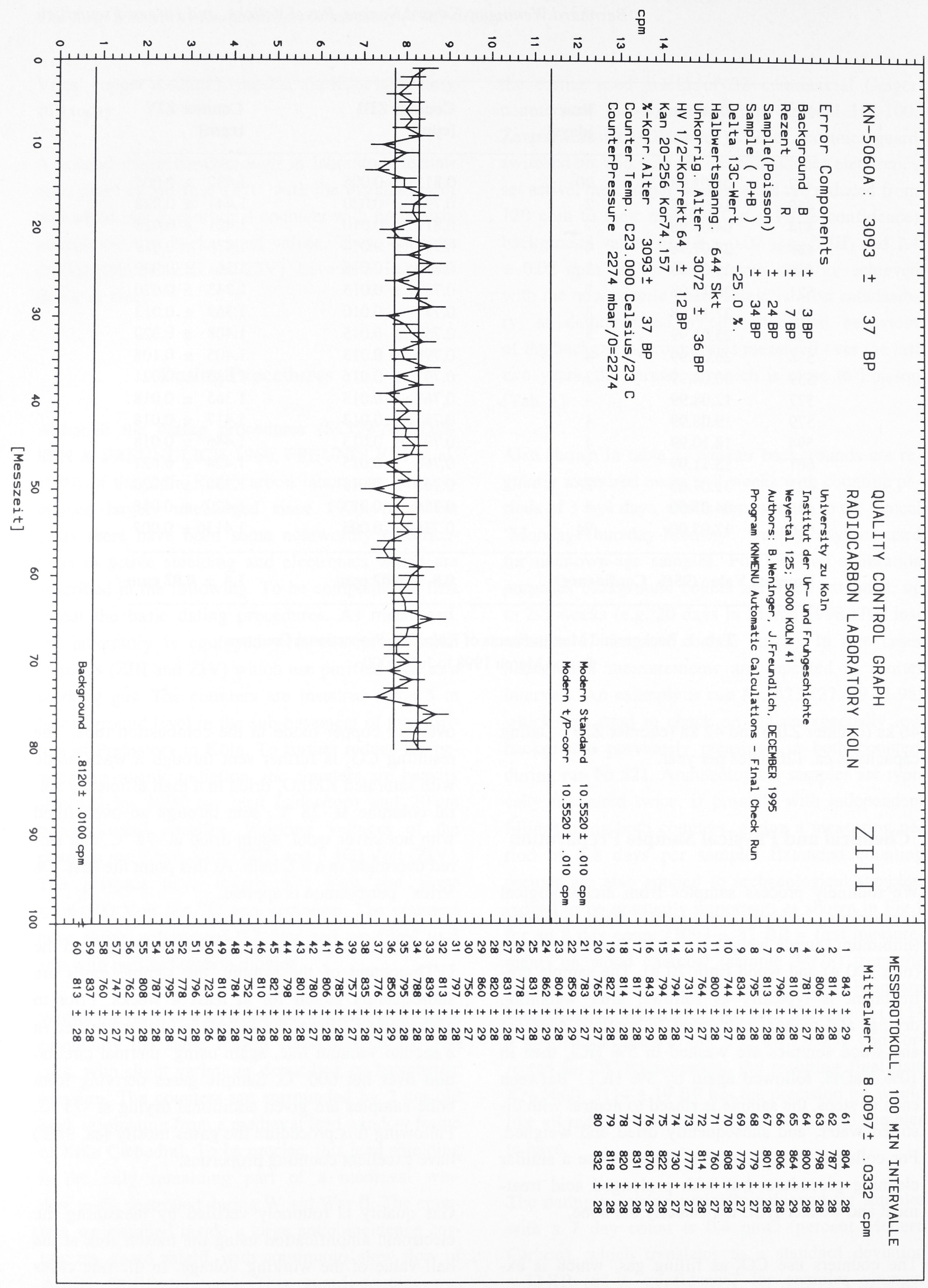


**Fig. 1** Quality control graph, showing the $^{14}C$ counts for wood sample KN-5060A, as measured in counter ZIII with 8000 minute run. Counts are listed at 100 minute intervals. The graph shows the error bars (± 1$\sigma$) as calculated for a Poisson distribution of decay events during each 100 min interval, in comparison to the theoretical (68%) Poisson limits (shown as lines).

| Lab. Nr<br>(1) | $^{14}$C-Age<br>[BP±1$\sigma$]<br>(2) | $\delta^{13}$C<br>[‰]<br>(3) | Ring<br>Nr<br>(4) | Ring<br>Nr<br>(5) | Centre<br>Ring<br>(6) | DendroAge<br>[denBC]<br>(7) |
|---|---|---|---|---|---|---|
| KN-4547 | 6197 ± 35 | -26.97 | 129 | -- | 5095 | 5098-5092 |
| KN-5110 | 6249 ± 48 | -27.11 | -- | 220 | 5098 | 5094-5103 |
| KN-4548 | 6260 ± 48 | -27.24 | 120 | -- | 5102 | 5106-5097 |
| KN-5111 | 6155 ± 62 | -26.87 | -- | 210 | 5109 | 5104-5113 |
| KN-4549 | 6240 ± 46 | -26.62 | 110 | -- | 5112 | 5116-5107 |
| KN-5112 | 6192 ± 35 | -26.95 | -- | 200 | 5118 | 5114-5123 |
| KN-4550 | 6178 ± 44 | -26.67 | 100 | -- | 5122 | 5126-5117 |
| KN-5113 | 6184 ± 35 | -26.77 | -- | 190 | 5130 | 5124-5133 |
| KN-4551 | 6219 ± 37 | -25.71 | 90 | -- | 5132 | 5136-5127 |
| KN-5114 | 6211 ± 48 | -26.02 | -- | 180 | 5139 | 5134-5143 |
| KN-4552 | 6211 ± 36 | -25.97 | 80 | -- | 5142 | 5146-5137 |
| KN-5115 | 6294 ± 30 | -26.91 | -- | 170 | 5149 | 5144-5153 |
| KN-4553 | 6281 ± 44 | -26.19 | 70 | -- | 5152 | 5156-5147 |
| KN-5116 | 6236 ± 60 | -26.07 | -- | 160 | 5160 | 5154-5163 |
| KN-4554 | 6317 ± 41 | -26.73 | 60 | -- | 5162 | 5166-5147 |
| KN-5117 | 6254 ± 45 | -26.47 | -- | 150 | 5168 | 5164-5173 |
| KN-4555 | 6186 ± 46 | -26.57 | 50 | -- | 5172 | 5176-5157 |
| KN-5118 | 6205 ± 34 | -27.42 | -- | 140 | 5175 | 5174-5183 |
| KN-4556 | 6115 ± 36 | -26.25 | 40 | -- | 5182 | 5186-5167 |
| KN-5119 | 6090 ± 45 | -27.03 | -- | 130 | 5189 | 5184-5193 |
| KN-4557 | 6083 ± 34 | -26.20 | 30 | -- | 5192 | 5196-5177 |
| KN-5120 | 6057 ± 35 | -27.19 | -- | 120 | 5199 | 5194-5203 |
| KN-4558 | 6199 ± 39 | -26.58 | 20 | -- | 5202 | 5206-5187 |
| KN-5121 | 6258 ± 45 | -26.54 | -- | 110 | 5209 | 5204-5213 |
| KN-4559 | 6223 ± 51 | -26.61 | 10 | -- | 5212 | 5216-5197 |
| KN-5122 | 6262 ± 46 | in prep. | -- | 100 | 5219 | 5214-5123 |
| KN-4560 | 6187 ± 36 | -26.83 | 2 | -- | 5222 | 5226-5207 |
| KN-5123 | 6233 ± 48 | -26.67 | -- | 90 | 5229 | 5224-5233 |
| KN-5124 | in prep. | in prep. | -- | 80 | 5239 | 5234-5243 |
| KN-5125 | 6472 ± 44 ( ?) | -26.78 | -- | 70 | 5251 | 5244-5253 |
| KN-5126 | 6229 ± 49 | -26.92 | -- | 60 | 5260 | 5254-5263 |
| KN-5127 | 6327 ± 46 | -27.29 | -- | 50 | 5269 | 5264-5273 |
| KN-5128 | 6162 ± 31 | -26.99 | -- | 40 | 5280 | 5274-5283 |
| KN-5129 | 6191 ± 31 | -26.95 | -- | 30 | 5290 | 5284-5293 |
| KN-5130 | 6290 ± 38 | -27.52 | -- | 20 | 5299 | 5294-5303 |
| KN-5131 | 6231 ± 48 | -27.61 | -- | 10 | 5309 | 5304-5313 |
| KN-5132 | 6425 ± 50 | -28.13 | -- | 0 | 5316 | 5314-5323 |

**References**

(1) Köln Laboratory Number (sample identifier).
(2) $^{14}$C-Age [yrs BP], ref. Oxalic Acid NBS, $T_{½}$ = 5568 [BP].
(3) $^{13}C/^{12}C$-correction, $\sigma$ = ± 0.01 ‰
(4) Tree ring Nr of wood sample Nr.85 (Dendro-Lab Number).
(5) Tree ring Nr of wood sample X-353a (Dendro-Lab Number).
(6) Approximate (± 2 yr) Dendro Age of central tree-ring of sample.
(7) Dendro Ages of all rings (different width) covered in the sample.

**Tab. 2** $^{14}$C-Dates for two oak logs from the wooden-box construction no. 2 of the Early Neolithic Well at Erkelenz-Kückhoven (Köln, Germany).

for at least two further days, with additional drying. In our routine work, variations in gas purity are always within ± 30 volts (ZIII) or ± 10 volts (ZIV) of the standard working voltage. This allows application of constant high voltage to all samples (ZIII: 7000 volt; ZIV: 5100 volt), and count-rate cor-

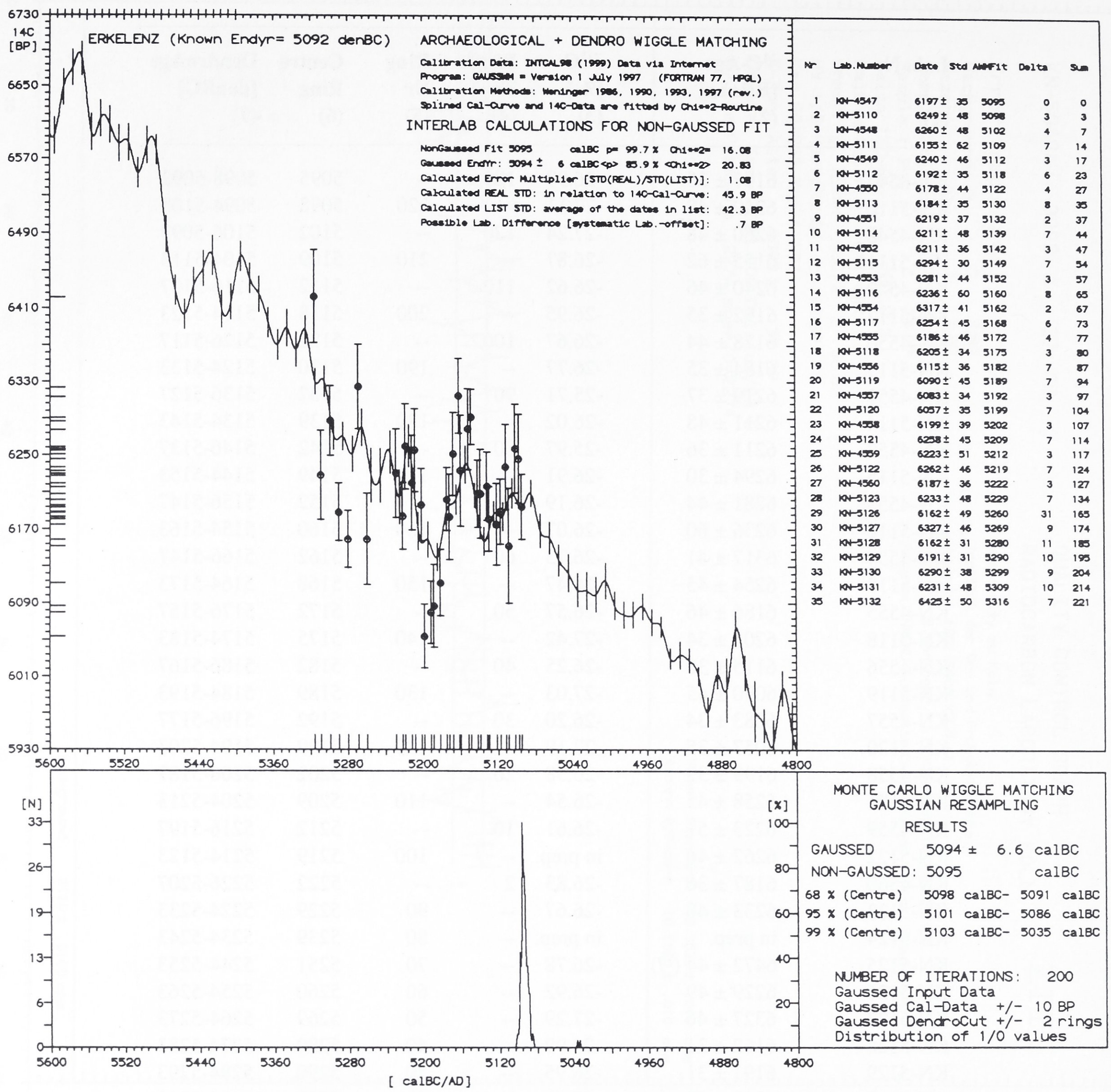


**Fig. 2** Application of GMCWM (Gaussian Monte Carlo Wiggle Matching) to the $^{14}$C-series from Erkelenz-Kückhoven (Tab. 2), showing that the trees used in the construction were cut (2 rings after: see below) the year 5095 calBC (best-fit single age) resp. 5094 ± 7 calBC (best-fit age distribution). The age distribution obtained by the GMCWM method allows for the following error components 1) wood cutting errors of ± 2 rings, 2) dynamic cal-curve/interlab errors of ± 10 $^{14}$C-BP, 3) standard errors of input data as given in Tab. 2 (N = 34, one outlier deleted). The youngest sample contains 4 rings. The GMCWM result is referenced to the middle of the youngest sample. Because we are interested in the outer ring = cutting year, the calculated result ("Gaussed 5094 ± 6.6 calBC") needs an off-line correction of 2 rings to the younger. The corrected (and finally rounded) result (5092 ± 7 cal BC) is identical to the measured dendro-age (5092 denBC: WEINER 1998) for the box construction No. 2.

rections resulting from remaining slight variations in gas purity are based on an empirical relation between the shape of the $^{14}$C beta-decay energy loss spectrum and the beta count rate for pure and less pure gases. This procedure allows correction of any time-variable change in gas-purity during the counting process, which makes the correction more reliable than simply measuring the half-voltage to begin and end of the specific run, but which we also monitor for control-purposes.

| **Lab.Nr** (1) | **$^{14}$C-Age [BP±1$\sigma$]** (2) | **$\delta^{13}$C [‰]** (3) | **Köln Lab.** (4) | **Belfast Lab.** (5) | **Seattle Lab.** (6) | **Tree Age** (7) |
|---|---|---|---|---|---|---|
| KN-2431 | 3297 ± 71 | -24.83 | | | | |
| | 3322 ± 69 | " | | | | |
| | 3265 ± 53 | " | | | | |
| | 3283 ± 52 | " | | | | |
| | 3311 ± 48 | " | | | | |
| **Average:** | **3294 ± 25** | | **3286 ± 27** | **3286 ± 14** | **3275± 27** | **1589 denBC** |
| KN-2438 | 3169 ± 55 | -24.39 | | | | |
| | 3181 ± 25 | " | | | | |
| | 3254 ± 49 | " | | | | |
| **Average:** | **3202 ± 21** | | **3181 ± 28** | **3198 ± 10** | **3178 ± 18** | **1449 denBC** |
| KN-2440 | 3124 ± 74 | -24.46 | | | | |
| | 3056 ± 60 | " | | | | |
| | 3112 ± 51 | " | | | | |
| **Average:** | **3116 ± 34** | | **3108 ± 27** | **3097 ± 14** | **3090 ± 26** | **1409 denBC** |

**References**
(1) Köln Laboratory Number (sample identifier).
(2) $^{14}$C-Age [yrs BP], ref. Oxalic Acid NBS, $T_{1/2}$ = 5568 [BP].
(3) $^{13}$C/$^{12}$C-Correction, $\sigma = \pm 0.01$ ‰
(4) Köln Laboratory Result on Identical Sample Gas (FREUNDLICH 1984)
(5) Belfast Laboratory Result of Wood of Identical (± 10 a) Age .
(6) Seattle Laboratory Result of Wood of Identical (± 10 a) Age.
(7) Dendro Sample Age

**Tab. 3** $^{14}$C-Ages on Wood Samples from Ipweger Moor. Recent measurements and weighted averages (1993-2000).

Due to the prohibitively low count rates, we have not yet been able to measure a dependency of background count rates on gas purity, which is the expected result for ionization events starting from/near the inner counter surfaces (i.e. not from the central volume). The counter backgrounds show no (sensibly correctable) dependence on barometric pressure changes.

## Quality Control

A crucial question for any radiocarbon dating laboratory, once the equipment has been precisely calibrated, is to maintain the established precision over extended time-periods of years. This is important, especially in archaeological studies, because 1) the radiocarbon method is destructive, 2) many samples are unique historic documents, 3) the sampling process (excavation) is expensive and 4) the excavation can seldom be repeated. As a consequence, the dating of archaeological samples can seldom be repeated and the dates thus themselves become unique historic documents. All these aspects call for a reliable, systematic, and continuous quality control for $^{14}$C dates on archaeological samples. [We note, en passim, that all this is quite nice to write, but slightly more difficult to maintain. In actual fact, in our laboratory, for the first time in the past 30 years we have recently lost a bone sample. That is a human scull. It has become the laboratory ghost. Its existence is proven by vast amounts of sequentially interrelated purposely self-controlling laboratory documents, all of which follow each and every sample through each and every step in the sample processing. The scull itself is nowhere to be found.]

## Interlaboratory Calibration

A major question in radiocarbon dating is the precision and accuracy of the equipment modern calibration. For equipment calibration, we use (only) the primary NBS oxalic acid. However, due to the rather low $^{14}C$ decay rates of the oxalic acid standard, the equipment calibration is supplemented by measurements using the Heidelberg 'Wilhelm' secondary standard. This sample gives a factor 10 higher count rate than the oxalic acid, which greatly helps reduce time e.g. when measuring the shape and length of the $^{14}C$ plateau and/or the gas purity correction curve.

On the base of earlier studies, utilizing 25 $^{14}C$-measurements on decadel wood samples from the Ipweger Moor bog courseway (FREUNDLICH 1984; MANNING & WENINGER 1992), it has been shown that the modern calibration of the Köln $^{14}C$ data is in accordance with data from most other laboratories, to within ± 20 $^{14}C$-yrs (95% confidence), depending on the comparison laboratory. In this question we must be specific. For example, $^{14}C$ data from Köln (FREUNDLICH 1984) show an (insignificant) offset of -3.4 $^{14}C$ yrs in comparison to data from Seattle (1976). However, such small deviations in equipment calibration are more difficult to evaluate than often acknowledged, mainly because samples of the 'same' $^{14}C$-age are quite difficult to procure and also because precise measurements are extremely time-consumung, at least for laboratories using the traditional $^{14}C$ decay technique. Thus, to be precise, it appears that a routinely dated set of radiocarbon dates, produced in the Köln laboratory in the years 1970 to 1972, agrees well (within ± 10 $^{14}C$-yrs) with dates produced in the Belfast and Seattle laboratories, and published in INTCAL86, that are measured on wood samples of the same calendric age but of different provenience (North German Oak vs Irish Oak). We must abbreviate on these difficulties. Important is that the Köln modern standard has remained unchanged in all the years 1970 to 1992, after which the new electronic data acquisition system made a recalibration of the equipment necessary. This is useful to know, because Köln data are typically used in age-calibrated archaeological radiocarbon chronologies, and Belfast and Seattle are two of the major laboratories engaged in construction of the dendrochronological calibration curve.

Table 3 shows more recent (1992-2000) age comparisons of Köln and Belfast/Seattle data. Again, the agreement is quite satisfactory. More precise estimates have recently become available, now that we have nearly completed radiocarbon dating of the Early Neolithic Well of Erkelenz-Kückhoven (WEINER 1998). The numeric results are given in table 2, with a preliminary analysis based on the method of Gaussian Monte Carlo Matching (JÖRIS & WENINGER 2000) shown in figure 2. With the help of these dates, we estimate that the modern calibration of the Köln dating equipment has not changed measurably (to within ± 10 BP), following the laboratory modernisation.

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