

Analysis of Uncertainty Budgets for $4\pi\beta\text{-}\gamma$ Coincidence Counting: a Simple Comparison Exercise.

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1 Introduction

In order to better understand the discrepancies observed in the uncertainties reported in $4\pi\beta\text{-}\gamma$ coincidence activity measurements, the CCRI (II) Uncertainties Working Group UCWG (II) proposed that a simple comparison exercise be undertaken to examine how various NMIs determine and report uncertainties entailed by the coincidence counting technique for radionuclide standardization.

This comparison will focus on two of the dominant uncertainty components commonly quoted for $4\pi\beta\text{-}\gamma$ coincidence counting in SIR submissions and Key Comparison exercises: namely, efficiency-extrapolation and weighing. Restricting the scope of the comparison to these two components makes sense because doing otherwise would make the analysis of a single data set intractable since NMIs use different types of deadtimes, live-timing techniques and coincidence count rate corrections.

However, as a subsidiary exercise, participants are encouraged to *describe the methods* they would typically use to evaluate the remaining uncertainty components; they may also choose to estimate the values of these components if they wish to do so.

The data set provided for this comparison pertains to a ^{60}Co solution which was standardized at NPL in December 2008. The solution was used to prepare a set of ten VYNS sources, which were then measured for a week by means of NPL's $4\pi\beta\text{-}\gamma$ coincidence system. An ampoule prepared from the same stock solution was later submitted to the SIR, in August 2009.

Section 2 of this document presents the source preparation procedure and the relevant weighing data for calculating the masses of the ten sources. The coincidence counting set-up and counting data are provided in section 3. An appendix at the end of this document contains additional data which may be of interest or use for estimating the uncertainties.

2 Source preparation and weighing data

2.1 Source preparation

The ^{60}Co radioactive solution used for preparing the sources was in hydrochloric form (0.1 M) with a CoCl_2 carrier concentration of 100 $\mu\text{g/g}$. A 24-hour gamma spectrometry measurement revealed no gamma emitting impurities.

A series of 10 VYNS sources, denoted S08078... S08087, were prepared by drop dispensation using the pycnometer method. The droplets deposited on the VYNS supports were subsequently desiccated with an IRMM source dryer.* This was done on 24 November 2008.

The balance used for weighing the pycnometer was a Mettler AT20. Before each weighing, the pycnometer was exposed to a static charge eliminator in order to minimise the effects of electrostatic charge build-up on it.

The weighing data was recorded in such a manner as to allow the use of the "direct weighing" method or the "substitution weighing" method.

* Denecke *et al*, Appl. Radiat. Isot. **52** (2000) 351-355.

In the “direct” method of weighing, for each source prepared, the balance reading was recorded before and after each deposition. The zero load (or “tare”) readings were also taken before and after each insertion of the pycnometer into the balance. The mean of the tare readings encompassing each pycnometer reading was subtracted from that pycnometer reading, in order to compensate for any drift in the balance. The difference between the drift[†]-corrected masses of the pycnometer, *before* and *after* the deposition, was taken as the mass of the droplet W_{direct} (uncorrected for buoyancy).

In the “substitution” method, a set of traceable calibration weights – see their certificate of calibration in appendix 5.1 – were immediately placed on the balance with the sum of their nominal masses (W_2) chosen to be as close as possible to the recorded reading of the recently removed pycnometer mass (W_1). If the recorded mass of the sum of these calibration weights is denoted by W_3 , the mass of the pycnometer W (uncorrected for buoyancy) was calculated as:

$$W = (W_2 + W_1 - W_3).$$

The aliquot deposited on any VYNS support (uncorrected for buoyancy) was determined as the difference between the calculated masses of the pycnometer, *before* and *after* the deposition, i.e.

$$W_{\text{subst}} = (W_2 + W_1 - W_3)_{\text{before deposition}} - (W_2 + W_1 - W_3)_{\text{after deposition}}$$

2.2 Weighing data

2.2.1 Data for direct reading weighing

Table 1 below reports the zero load (or tare) readings before and after the pycnometer is placed onto the balance as well as the pycnometer mass readings before and after dispensing a drop of the solution onto the VYNS support of each source.

Table 1

Source ID	tare before [g]	Pycno. mass before deposition [g]	tare in-between [g]	Pycno. mass after deposition [g]	tare after [g]
S08078	0.000010	3.633470	0.000010	3.617694	0.000008
S08079	0.000030	3.617808	0.000032	3.602942	0.000034
S08080	0.000058	3.602952	0.000062	3.587710	0.000060
S08081	0.000086	3.587752	0.000086	3.572610	0.000092
S08082	0.000102	3.572646	0.000106	3.556804	0.000106
S08083	0.000128	3.556846	0.000130	3.540768	0.000136
S08084	0.000152	3.540820	0.000158	3.525160	0.000160
S08085	0.000180	3.525192	0.000186	3.491290	0.000188
S08086	0.000210	3.491350	0.000220	3.458518	0.000220
S08087	0.000250	3.458562	0.000254	3.426846	0.000258

[†] Balances drift because of the heat gradients and air turbulence in the laboratory and the convection and electrostatic charging inside the balance.

2.2.2 Data for substitution weighing

Table 2 below reports, from left to right, the pycnometer mass reading (W_1), the list of calibration substitution weights which have a total nominal mass as close as possible to W_1 , the value of this total nominal mass (W_2) and the actually recorded mass of the sum of these calibration weights (W_3). The table lists these values *before* and *after* dispensing a drop of the solution onto the VYNS support of each source.

The certificate of the traceable calibration weights {C30, ..., C53} is provided in appendix 5.1.

Table 2

	<i>Before pycnometer drop deposition</i>				<i>After pycnometer drop deposition</i>			
Source ID	W_1 [g]	Substitution set	W_2 [g]	W_3 [g]	W_1 [g]	Substitution set	W_2 [g]	W_3 [g]
S08078	3.633470	C38+C40+C41+C44+C46+C48+C50+C52	3.632995	3.633032	3.617694	C38+C40+C41+C44+C48+C49+C50	3.616990	3.617042
S08079	3.617808	C38+C40+C41+C44+C48+C49+C50	3.616990	3.617052	3.602942	C38+C40+C41+C44+C50+C52	3.602987	3.603048
S08080	3.602952	C38+C40+C41+C44+C50+C52	3.602987	3.603056	3.587710	C38+C40+C41+C45+C46+C48+C49+C50	3.587001	3.587082
S08081	3.587752	C38+C40+C41+C45+C46+C48+C49+C50	3.587001	3.587104	3.572610	C38+C40+C41+C45+C46+C50	3.571996	3.572104
S08082	3.572646	C38+C40+C41+C45+C46+C50	3.571996	3.572118	3.556804	C38+C40+C41+C45+C49+C52	3.555994	3.556124
S08083	3.556846	C38+C40+C41+C45+C49+C52	3.555994	3.556138	3.540768	C38+C40+C41+C46+C47+C52	3.540989	3.541140
S08084	3.540820	C38+C40+C41+C46+C47+C52	3.540989	3.541158	3.525160	C38+C40+C41+C46+C49	3.524992	3.525164
S08085	3.525192	C38+C40+C41+C46+C49	3.524992	3.525192	3.491290	C38+C40+C42+C43+C45+C46+C47+C52	3.491017	3.491226
S08086	3.491350	C38+C40+C42+C43+C45+C46+C47+C52	3.491017	3.491256	3.458518	C38+C40+C42+C43+C45+C49+C50+C52	3.458020	3.458262
S08087	3.458562	C38+C40+C42+C43+C45+C49+C50+C52	3.458020	3.458294	3.426846	C38+C40+C42+C43+C46+C49+C52	3.426019	3.426302

2.2.3 Balance data

The balance used for weighing the pycnometer was a Mettler AT20. The balance had been calibrated on 8 May 2008. The certificate of calibration of the balance is provided in appendix 5.2. Details about the repeatability, off-centre loading, linearity and uncertainty of its measurements are provided in the certificate.

Note that the uncertainty stated in the certificate is not expressed in a standard way. For instance, NMIs which have their balances periodically checked by Mettler Toledo's calibration laboratory get a certificate in which the uncertainty is not constant but rather variable over the weighing range, often proportional to the load.

Using the AT20's data provided in the certificate of calibration in appendix 5.2, the procedure DKD-R 7-1 was used to evaluate the weighing uncertainty. The absolute standard uncertainty of a given weighing was found to be $u = 3.14 \cdot 10^{-6} + W \cdot 6.568 \cdot 10^{-6}$ [g], in which W represents the reading in grams for the given load. Participants may use either the uncertainty stated in the certificate or this algorithm.

About six months had elapsed between the calibration of the balance and the preparation of the VYNS sources (24 November 2008). During this period the AT20 continued to be monitored. Two weights, of nominal mass 2g and 20g respectively, were measured (with zero-load readings before and after) several times per week. The masses of the weights were logged as the balance reading minus the mean of the zero-load readings preceding and following their weighing. This data is available in appendix 5.3.

2.2.4 Buoyancy correction data

The mass measurements were not performed in vacuum, so both the solution and the calibrations weights are affected by the buoyancy in the ambient air. Appendix 5.4 describes how the buoyancy correction may be performed.

The ambient temperature T (in °C), the atmospheric pressure P (in hPa) and the relative humidity H (in percent) were logged at the time of drop deposition. Table 3 reports these parameters for each source prepared. The certificate of calibration for the temperature, pressure and humidity logger is given in appendix 5.5.

Table 3

Source ID	Temp. [°C]	Press. [hPa]	Rel Hum. [%]	Buoyancy correction
S08078	21.40	1000.7	28.9	1.001032
S08079	21.90	1001.5	37.2	1.001030
S08080	21.90	1001.8	38.3	1.001030
S08081	22.00	1001.9	40.3	1.001030
S08082	22.50	1002.1	41.4	1.001028
S08083	22.10	1002.3	42.4	1.001029
S08084	22.20	1002.2	42.2	1.001029
S08085	22.20	1002.3	43.1	1.001029
S08086	22.20	1002.4	43.9	1.001029
S08087	22.20	1002.8	45.1	1.001029

The buoyancy correction is computed in the last column using the prescription in appendix 5.4. This was calculated with a density of $1002(1) \text{ kg}\cdot\text{m}^{-3}$ ($k=1$) for the ^{60}Co radioactive solution and a density of $8000(200) \text{ kg}\cdot\text{m}^{-3}$ ($k = 1$) for the calibration weights.

2.2.5 Other weighing data

Some NMIs use the full calibration history of the balance in estimating the uncertainty of its weighing. This data shall not be provided in this exercise as it would be cumbersome to collect and dispatch all the certificates of the balance.

No data is available about evaporation during this source preparation. It is up to NMIs to use their own numbers, estimated from their own practical experience, if they wish to include this contribution to the uncertainty of the masses of the sources.

3 Coincidence counting system and data

3.1 Counting system

The set of these ^{60}Co sources were measured on NPL's coincidence counting system shown below.

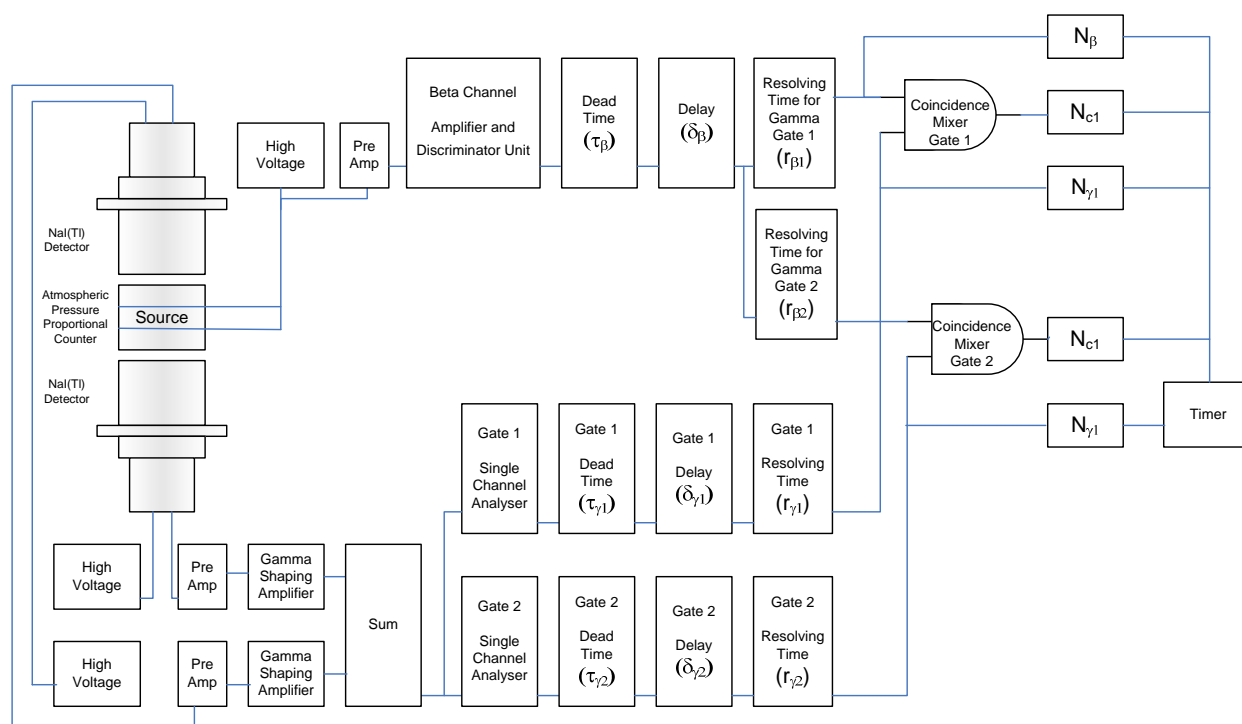


Figure 1: Experimental set-up for $4\pi\beta\text{-}\gamma$ coincidence counting at NPL

The beta counter used is a proportional counter employing P-10 gas, operated at atmospheric pressure. Gamma detection involves the summation of the outputs from two NaI(Tl) detectors. Each NaI(Tl) detector was of the Type Harshaw 16MB16/3A, which corresponds to a size of 4" by 4" \varnothing .

3.2 Counting conditions

In the beta channel, the counter was operated at 1850 V, well within its plateau region. More details about its plateau characterisation are available in appendix 5.6.

NPL's "in-house" beta channel amplifier unit combines the functions of signal amplification of input pulses from the pre-amplifier, imposition of a discriminator level (on a differentiated signal) and imposition of a non-extendable dead time of desired length. The beta discriminator level was set to just above the electronic noise. The beta deadtime (τ_β), which is of a non-extending type, was measured to be 10.1 (1) μs ($k=1$). More information about this deadtime measurement and the behaviour of the discriminator/dead-time circuitry is provided in appendix 5.7. Note that no pile-up rejector was employed, as the deadtime is effectively triggered by the derivative of the input pulse.

The beta detection efficiency was varied using attenuation with gold-coated VYNS foils and aluminium foils. The intrinsic VYNS thickness was about 30 $\mu\text{g}\cdot\text{cm}^{-2}$ while that of the gold was approximately 10 $\mu\text{g}\cdot\text{cm}^{-2}$. The aluminium foils had a thickness of 200 $\mu\text{g}\cdot\text{cm}^{-2}$. The transition from foiling with gold-coated VYNS to the use of aluminium foils was triggered when the beta efficiency decreased to approximately 83%.

The maximum beta efficiency thus obtained was 93.8 % and its minimum was 75.4 %.

As pointed out earlier, the gamma channel is comprised of the summation of the outputs from two NaI(Tl) detectors. The figure below shows the spectra obtained from each of the single detectors (red and blue points) as well as the summed spectrum (black points).

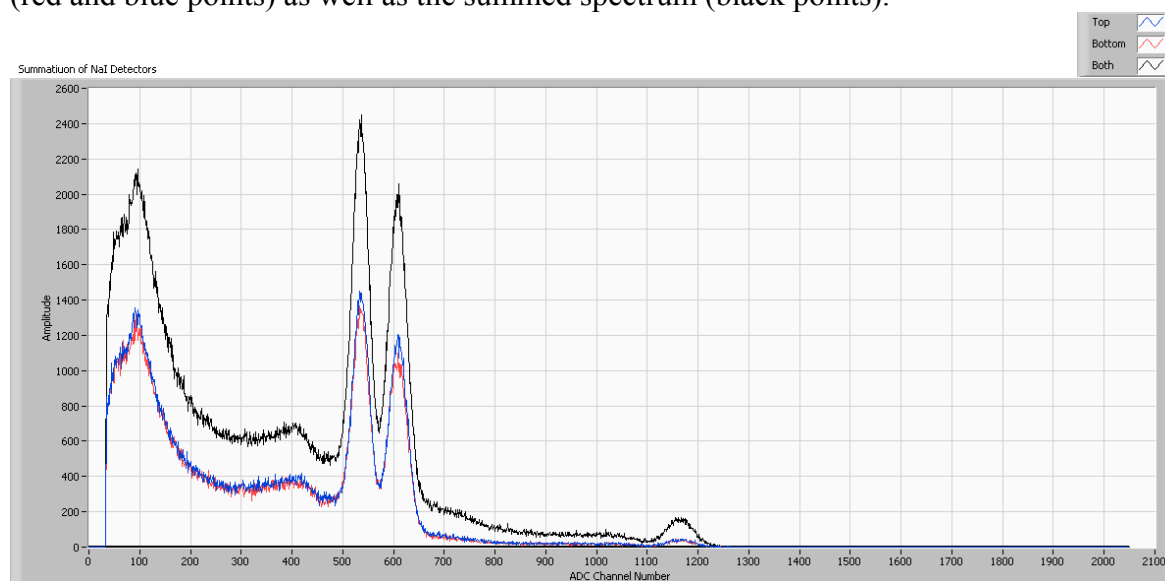


Figure 2: Typical γ -channel spectrum

For this exercise, two gamma gates were selected on the summed spectrum: the first denoted as "Gamma gate 1" was selected to encompass only the photo-peaks from the 1173 and 1332 keV gamma transitions.

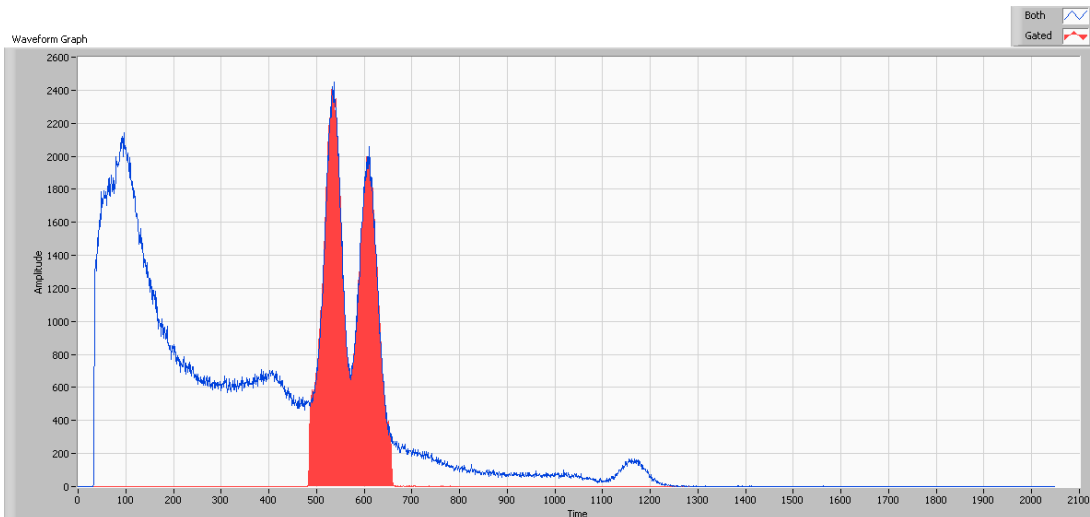


Figure 2: Gamma gate 1

The second gamma-energy condition, referred to as “Gamma gate 2”, was a threshold set at approximately 90 keV.

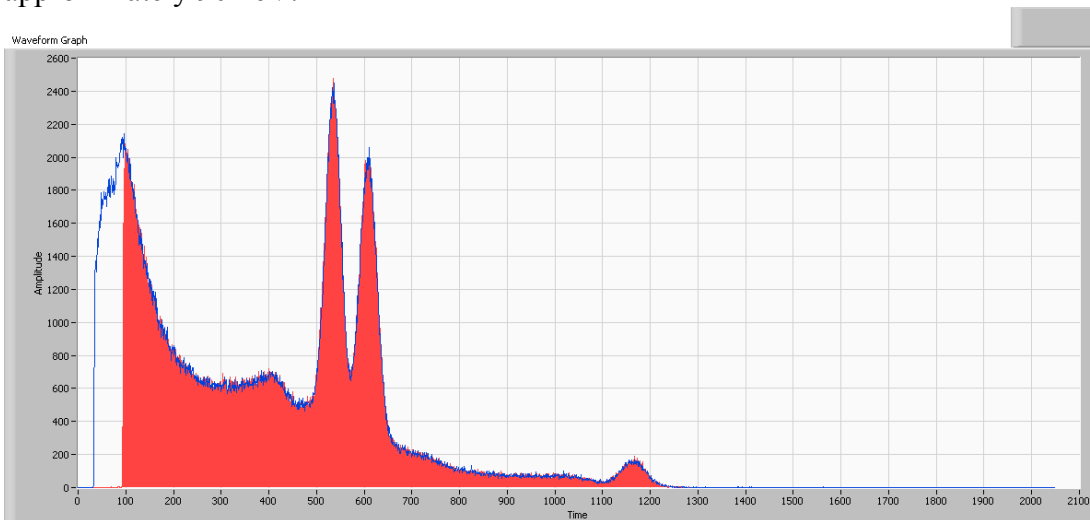


Figure 3: Gamma gate 2

The gamma channel deadtime (τ_γ) is nonparalyzable. Dual pulser measurements yielded a deadtime of 10.0 (5) μs ($k = 1$) for gamma gate 1, and 15.1 (5) μs ($k = 1$) for gamma gate 2. Appendix 5.8 gives the particulars of this deadtime measurement. Note that no pile-up rejector was employed in the gamma channel.

The average gamma detection efficiency was found to be 13.7 % for gamma gate 1, and 37.5 % for gamma gate 2.

With regards to the coincidence channel, the beta channel coincidence resolving time (i.e. the width of the pulses sent to the appropriate coincidence mixer) was measured to be 0.50(1) μs ($k=1$) in the case of gamma gate 1 ($r_{\beta 1}$) and 0.70(1) μs ($k=1$) in the case of gamma gate 2 ($r_{\beta 2}$). The gamma channel coincidence resolving time for gamma gate 2, being the width of the pulses sent to the

relevant coincidence mixer, were $r_{\gamma_1} = 0.50(1) \mu\text{s}$ ($k=1$) and $r_{\gamma_2} = 0.70(1) \mu\text{s}$ ($k=1$). Appendix 5.9 explains how they were measured.

A final counting condition of interest is the property of the timer used. Each channel of the counter-timer card was fed a traceable 1 MHz signal for repeated counting periods of 10 seconds. The mean total number of observed counts was 10,000,020. Thus the frequency of the oscillator on the counter-timer was deemed to be adequately set, and the bias attributed to timing of counting periods was regarded as being 0.0002 %, and was thus deemed negligible.

3.3 Counting data

The measurement campaign lasted one week. The first measurement started on 12 December 2008 at 11:50:49 UTC while the last one ran on 19 December 2008 at 16:52:58 UTC. The reference time was set to 12:00:00 UTC 1st December 2008. The half-life of ^{60}Co was assumed to be 1925.2(3) days ($k=1$).

The measurement of every individual source, with or without attenuator and in any given gamma energy setting, consisted of a series of five counting cycles lasting 200 seconds each. This counting period was chosen so that for the weaker sources, the theoretical standard deviation of the mean for the estimates of ρ_β and ε_β were both less than 0.05%.

The acquisition of data from the coincidence system detailed in 3.1 was performed through NPL's BGC – Beta-Gamma-Coincidence – program, which provides interfacing between the NPL “OMEGA” scaler/timer card and a PC. The software can handle up to 3 gamma gates concurrently (although only two were used in this exercise). A sample output from the BGC program, for a single source measurement, is shown, for illustration, in appendix 5.10.

The file named Co60_ComparisonData.xls sent to the participants contains a summary of the data reported by the program BGC. There are individual Excel sheets for sources S08079 up to S08084 whereas the counting data of the high activity sources S08085, S08086 and S08087 are compiled within a single worksheet. Two additional Excel sheets, in this file, combine all the data for gamma gate 1 and for gamma gate 2. This file contains all the necessary data required for your efficiency extrapolation fitting routines.

Consider the Excel worksheet for a particular source, say S08079. The first two columns, from the left, identify the source and the attenuator foils used if any. The table below explains the nomenclature used for the foils.

Designation	Refers to
1T	one VYNS foil on the top of the source
1T1B	one VYNS foil added to bottom of existing 1T
2T1B	one VYNS foil added to the top of existing 1T1B
5T5B	five VYNS foils on the top and five on the bottom of the source
Al_1T	one aluminium foil on the top (i.e.: added on top of the existing 5T5B)
Al_1T1B	one aluminium foil on the bottom (i.e.: on top of the existing 5T5B_Al_1T)

The third column is entitled b/eb; b is shorthand for ρ_β , the estimate of the true beta countrate at the reference time, while eb stands for ε_β , the estimate of the beta detection efficiency. Hence b/eb denotes $\rho_\beta/\varepsilon_\beta$, which is equivalent to $\rho_\beta \cdot \rho_\gamma / \rho_c$ – the estimate of the apparent disintegration rate – where ρ_γ and ρ_c correspond to the estimates of the true gamma and coincidence rates, at the reference time, respectively.

Note, however, that the countrates listed in this file are not expressed in cps but in cps/mg, that is to say that b/eb refers in fact to $(\rho_\beta \cdot \rho_\gamma) / (m \cdot \rho_c)$ where m is the mass of source S08079. The values of the masses used for the ten sources were those obtained using the substitution weighing data; they are reported in appendix 5.11.

The fifth column tabs the values of $(1 - \varepsilon_\beta) / \varepsilon_\beta$ for those who wish to perform an efficiency extrapolation of the $\frac{\rho_\beta}{\varepsilon_\beta} = f\left(\frac{1 - \varepsilon_\beta}{\varepsilon_\beta}\right)$ type, while the fifth and sixth columns record values for use in a $\rho_\beta = f(1 - \varepsilon_\beta)$ extrapolation. The seventh column displays ε_β , and the eight column – entitled eg – lists the estimate of the gamma detection efficiencies (ε_γ).

Table 4 below summarises the notation used in the Excel file.

Table 4: notation used in the spreadsheet

b/eb	1/eb-1	b	1-eb	eb	eg
$\frac{\rho_\beta \rho_\gamma}{\rho_c}$	$\frac{1 - \varepsilon_\beta}{\varepsilon_\beta}$	ρ_β	$1 - \varepsilon_\beta$	ε_β	ε_γ

Note that the values listed in columns 3 to 8 are weighted means over the five cycles, using the inverse of the observed variances for the weighting factors.

The columns 9 to 15 of the S08079 worksheet report the *observed* percentage standard deviation of the mean (obs % sd) of the $N = 5$ cycle measurement for each of the quantities listed in columns 3 to 8. The last six columns list the *theoretical* percentage standard deviation of the mean (th % sd).

Table 5 makes explicit how these statistical quantities were calculated:

Table 5: statistical calculation details

weighted mean	observed variance of the mean	observed percentage standard deviation of mean	theoretical percentage standard deviation of mean
$\bar{x} = \frac{\sum_{i=1}^N w_i x_i}{\sum_{i=1}^N w_i}$ $w_i = 1/V(x_i)$	$V(\bar{x}) = \frac{\sum_{i=1}^N w_i (x_i - \bar{x})^2}{(N-1) \sum_{i=1}^N w_i}$	$\frac{\sqrt{V(\bar{x})}}{\bar{x}} \times 100 \%$	$\frac{\sqrt{\frac{1}{\sum_{i=1}^N w_i}}}{\bar{x}} \times 100 \%$

Please note that in the framework of the required exercise (section 4.2.1 of the reporting form), participants must fit $(\rho_\beta \cdot \rho_\gamma) / (m \cdot \rho_c)$ as a function of $(1 - \varepsilon_\beta) / \varepsilon_\beta$ using the values in the b/eb and 1/eb-1

columns respectively, which are provided in the Excel spreadsheet. The relevant uncertainties are those listed in the corresponding *observed* percentage standard deviation columns. Participants are required to use these data as is, no value or associated uncertainty may be discarded, normalized or re-evaluated. In section 4.2.2 of the reporting form, participants are free to use the extrapolation function of their choice and include or re-evaluate data points and their uncertainties as they see fit.

The BCG code does not normally output all the individual raw N_β , N_γ , and N_c counts or the true ρ_β and ρ_c countrates. Typical values have however been calculated, for the background, a low activity source and a high activity source, for possible use by participants. Table 6 displays the background counts and countrates in all the relevant channels.

Table 6: Typical background counts (in 200 seconds) and true countrates

Channel i:	β	γ -gate 1	coinc. 1	γ -gate 2	coinc. 2
\bar{N}_i	320	192	4	2745	91
ρ_i (cps)	1.600	0.960	0.020	13.728	0.455

The true beta and gamma countrates are the countrates corrected for deadtime while the coincidence countrates are corrected for deadtime and loss of coincidences and gains of accidental coincidences using the Cox-Isham formalism.

Table 7 and 8 present the same observables for a low activity source (s08080) and a high activity one (s08085).

Table 7: Typical background counts (in 200 seconds) and true countrates for a weak source

Channel i:	β	γ -gate 1	coinc. 1	γ -gate 2	coinc. 2
cycle 1	960798	145771	130897	394224	356227
cycle 2	959533	145353	130776	393952	356131
cycle 3	958024	145772	131160	393138	355481
cycle 4	958771	145604	130943	393464	355644
cycle 5	960546	146506	131734	395151	357042
\bar{N}_i	959534	145801	131102	393986	356105
ρ_i (cps)	5040.39	736.11	690.10	2016.59	1888.03

ρ_β and ρ_γ are the estimates of the instantaneous beta and gamma channel count rate at the reference time after dead-time and background corrections, while ρ_c are the estimates of the instantaneous coincidence channel count rates at the reference time, after dead-time, coincidence-resolving time and background corrections.

Table 8: Typical background counts (in 200 seconds) and true countrates for a strong source

Channel i :	β	γ -gate 1	coinc. 1	γ -gate 2	coinc. 2
cycle 1	1991486	317802	270216	835331	724569
cycle 2	1995500	319050	271655	837146	726277
cycle 3	1992663	317475	269985	836378	725769
cycle 4	1990948	317667	269902	836930	725613
cycle 5	1993244	318026	270638	836439	725751
\bar{N}_i	1992768	318004	270479	836445	725596
ρ_i (cps)	11077.47	1627.91	1510.45	4450.76	4118.91

Note that the countrates in tables 7 and 8 are not corrected for decay.

4 Queries about the Data

Any questions regarding the data sets may be directed to:

John Keightley (NPL)
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Radioactivity and Neutron Group
Hampton Road, Teddington, Middlesex
TW11 0LW
United Kingdom
Ph: +44 208 943 6398
Email: john.keightley@npl.co.uk

or

François Bochud (IRA).
Insitut Universitaire de Radiophysique Appliquée
UNIL – CHUV
Grand-Pré 1
CH-1007 Lausanne
Switzerland
Ph: +41 21 31 48 142
Email: francois.bochud@chuv.ch

5 Appendix

5.1 Certificate of calibration for weights used in “substitution method”



NATIONAL PHYSICAL LABORATORY
Teddington Middlesex UK TW11 0LW Telephone +44 20 8977 3222

Certificate of Calibration



SET OF WEIGHTS
73390 111

This certificate is issued in accordance with the laboratory accreditation requirements of the United Kingdom Accreditation Service. It provides traceability of measurement to recognised national standards, and to units of measurement realised at the National Physical Laboratory or other recognised national standards laboratories. This certificate may not be reproduced other than in full, except with the prior written approval of the issuing laboratory.

FOR	National Physical Laboratory Hampton Road Teddington Middlesex TW11 0LW For the attention of Mr J Keightley
DESCRIPTION	A set of 23 weights supplied by Mettler as follows: 200 gram to 1 gram - integral weights of non-magnetic stainless steel with an assumed density of $8\,000\text{ kg m}^{-3} \pm 200\text{ kg m}^{-3}$ 0.5 gram to 0.001 gram - weights of non-magnetic stainless steel wire with an assumed density of $8\,000\text{ kg m}^{-3} \pm 200\text{ kg m}^{-3}$
IDENTIFICATION	Box labelled 73390 111
DATE OF CALIBRATION	8 October 2008 to 17 October 2008

Reference: E08100145

Page 1 of 3

Date of issue: 27 October 2008

Signed:  (Authorised signatory)

Checked by: 

Name: Stuart Davidson for Managing Director

NATIONAL PHYSICAL LABORATORY

Continuation Sheet

SET OF WEIGHTS
73390 111

MEASUREMENTS

The conventional mass of each of these weights was determined by weighing in air using standards of known mass and density.

TRACEABILITY

The measured values are traceable via the UK National Standard of Mass to the International Prototype of the Kilogram.

RESULTS

The results of the measurements are given in the Table of Results. Each value given in the second column represents the conventional value of mass. For a weight taken at 20 °C, the conventional mass is the mass of a reference weight of density 8 000 kg m⁻³ which it balances in air of density 1.2 kg m⁻³. This basis is taken by convention in accordance with OIML D 28. When the density of air differs from 1.2 kg m⁻³ it may be necessary to apply a correction for this difference. It may also be necessary to make an allowance for the difference in the density of the weights from 8 000 kg m⁻³.

UNCERTAINTY

The uncertainty evaluation has been carried out in accordance with the ISO document 'Guide to the expression of uncertainty in measurement' and with UKAS requirements. The reported uncertainties given in the Table of Results are based on a standard uncertainty multiplied by a coverage factor $k=2$, providing a level of confidence of approximately 95%. The uncertainties quoted are commensurate with those required for the calibration of OIML Class E₂ weights.

Reference: E08100145

Page 2 of 3

Checked by: *M/John*

14/35/2017

NATIONAL PHYSICAL LABORATORY

Continuation Sheet

SET OF WEIGHTS
73390 111

TABLE OF RESULTS

Nominal value	Measured conventional mass	Estimated measurement uncertainty
g	g	± mg
200	199.999 87	0.10
200	200.000 22	0.10
100	99.999 948	0.05
50	49.999 994	0.03
20	20.000 019	0.02
20	20.000 031	0.02
10	9.999 994	0.01
5	4.999 965	0.01
2	2.000 004 3	0.005
2	1.999 982 6	0.005
1	0.999 983 3	0.005
0.5	0.499 999 5	0.005
□0.2	0.200 013 2	0.003
□0.2	0.200 010 7	0.003
0.1	0.099 996 3	0.003
0.05	0.050 003 7	0.003
□0.02	0.020 004 1	0.002
□0.02	0.019 995 1	0.002
0.01	0.010 004 1	0.002
0.005	0.005 001 0	0.002
□0.002	0.002 001 3	0.002
□0.002	0.002 003 6	0.002
0.001	0.001 002 2	0.002

The results and uncertainties quoted refer to values at the time of calibration and make no allowance for subsequent drift in the values of the weights.

Reference: E08100145

Page 3 of 3

Checked by: *M. M. M.*

1995-03-01-0007

The values of the last page of the certificate are reproduced in the table below.

Mass ID	Nominal value [g]	Certified value [g]	uncertainty [g]
C30	200	199.9998700	0.0001000
C31	200 D	200.0002200	0.0001000
C32	100	99.9999480	0.0000500
C33	50	49.9999940	0.0000300
C34	20	20.0000190	0.0000200
C35	20 D	20.0000310	0.0000200
C36	10	9.9999940	0.0000100
C37	5	4.9999650	0.0001000
C38	2	2.0000043	0.0000050
C39	2 D	1.9999826	0.0000050
C40	1	0.9999833	0.0000050
C41	0.5	0.4999995	0.0000050
C42	0.2	0.2000132	0.0000030
C43	0.2 D	0.2000107	0.0000030
C45	0.1	0.0999963	0.0000030
C46	0.05	0.0500037	0.0000030
C47	0.02	0.0200041	0.0000020
C48	0.02 D	0.0199951	0.0000020
C49	0.01	0.0100041	0.0000020
C50	0.005	0.0050010	0.0000020
C51	0.002	0.0020013	0.0000020
C52	0.002 D	0.0020036	0.0000020
C53	0.001	0.0010022	0.0000020

5.2 AT20 Balance: Certificate of Calibration

CERTIFICATE OF CALIBRATION	
ISSUED BY PRECISION BALANCE SERVICES LTD	CERTIFICATE NUMBER 91949
DATE OF ISSUE 30 May 2008	



P.B.S.
**PRECISION
 BALANCE
 SERVICES LTD.**
 Tel & Fax (01530) 834650

3 Atlas Court
 Atlas Road
 Hermitage Industrial Estate
 Coalville
 Leics LE67 3FL

Page 1 of 2 Pages
Approved Signatory: M.D.Exell Signature:

Customer National Physical Laboratory Hampton Road Teddington Middlesex TW11 OLW Named Contact Mr Sean Collins	Site National Physical Laboratory Hampton Road Teddington Middlesex TW11 OLW
---	---

Weighing Machine
Make Mettler **Model** AT 20 **Serial No** 1118110597

Range 1	Resolution	Range 2	Resolution	Range 3	Resolution	Range 4	Resolution
20g	0.000 002g						

Location G6-L15
Comments

Weight Sets Used 88M

The weighing equipment described above has been calibrated using weights traceable to National Standards and in accordance with the procedures in UKAS document LAB14 (where relevant). The results were recorded.

DEPARTURE FROM NOMINAL VALUE
 A series of weights were added to the centre of the load receptor. The reading at each load was recorded.

OFF-CENTRE ERRORS
 A load of between 1/4 and 1/3 the capacity of the machine was placed in the centre of the load receptor and the reading recorded. The load was then placed at each pan support in turn and again in the centre, the readings were recorded.

REPEATABILITY
 The load was applied to the centre of the load receptor and the reading recorded. The load was removed and the reading recorded. This was carried out ten times.

ENVIRONMENTAL MEASUREMENTS
 Environmental measurements will be made for weighing machines which have an accuracy greater than or equal to 0.01mg. Ambient temperature at the time of calibration will be recorded for all machines.

Results are reported in terms of conventional mass.
 For a weight taken at 20°C, the conventional mass is the mass of a reference weight of density of 8000kg.m⁻³ which it balances in air of a density 1.2kg.m⁻³.

This certificate is issued in accordance with the laboratory accreditation requirements of the United Kingdom Accreditation Service. It provides traceability of measurement to recognised national standards, and to the units of measurement realised at the National Physical Laboratory or other recognised national standards laboratories. This certificate may not be reproduced other than in full, except with the prior written approval of the issuing laboratory.

CERTIFICATE OF CALIBRATION

UKAS CALIBRATION LABORATORY No. 0459

Certificate Number

91949

Page 2 of 2 Pages

Balance Make: **Mettler**
Model: **AT 20**
Serial No: **1118110597**
Location: **G6-L15**
Range Calibrated: **20g x 0.000 002g**
Type of Calibration: **After Adjustment**
Internal Calibration Weight Activated: **Yes**

Date of Calibration: **08 May 2008**
Calibrator: **M.D.Exell**
Uncertainty of Measurements: **±0.000 071 1g**
Environmental Measurements
Temp: **20.0°C** RH: **48.7%** Air Pressure: **1002.0mbar**
Approved Signatory: *M D Exell*

Departure from Nominal Value

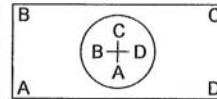
Applied Load (g)	Indicated Reading (g)
0.000 000	0.000 000
1.000 027	1.000 025
2.000 018	2.000 017
4.000 018	4.000 017
6.000 046	6.000 045
8.000 046	8.000 039
9.999 983	9.999 992
11.999 983	11.999 991
14.000 001	14.000 003
15.000 023	15.000 019
20.000 011	20.000 008

Repeatability Test Load Applied: 20.000 011g

Loaded Reading	Unloaded Reading	Difference
20.000 012	0.000 000	20.000 012
20.000 010	0.000 000	20.000 010
20.000 013	0.000 000	20.000 013
20.000 012	0.000 000	20.000 012
20.000 014	0.000 000	20.000 014
20.000 010	0.000 000	20.000 010
20.000 011	0.000 000	20.000 011
20.000 013	0.000 000	20.000 013
20.000 012	0.000 000	20.000 012
20.000 013	0.000 000	20.000 013
Range:		0.000 004g
Standard Deviation:		0.000 001 33

Eccentricity Test Load Applied: 5.000 019g

Ref Points	Indicated Reading
Centre	5.000 015
A	5.000 017
B	5.000 018
C	5.000 016
D	5.000 019
Centre	5.000 016



Departure from Nominal Value - As Found

Applied Load (g)	Indicated Reading (g)
0.100 007	0.100 011
2.000 000	2.000 012
5.000 019	5.000 016
9.999 983	10.000 002
20.000 011	20.000 014

The reported expanded uncertainty is based on a standard uncertainty multiplied by a coverage factor $k = 2$ providing a level of confidence of approximately 95%.

The uncertainty evaluation has been carried out in accordance with UKAS requirements.

5.3 Balance Repeatability

Date	Zero-load reading (g)	Nominal mass 2g	Zero-load reading (g)	Nominal mass 20g	Zero-load reading (g)	2g weight	20g weight
03/06/2008	0.000000	2.000012	0.000000	20.000024	0.000000	2.000012	20.000024
06/06/2008	0.000000	2.000010	0.000014	20.000040	0.000018	2.000003	20.000024
11/06/2008	0.000000	1.999996	-0.000002	20.000014	0.000000	1.999997	20.000015
12/06/2008	0.000000	2.000010	0.000000	20.000026	0.000010	2.000010	20.000021
16/06/2008	0.000000	1.999996	0.000000	20.000000	0.000002	1.999996	19.999999
18/06/2008	0.000000	2.000008	0.000010	20.000032	0.000030	2.000003	20.000012
19/06/2008	0.000000	2.000002	0.000000	20.000010	0.000000	2.000002	20.000010
20/06/2008	0.000000	2.000000	0.000004	20.000012	0.000000	1.999998	20.000010
23/06/2008	0.000000	2.000012	0.000000	20.000024	0.000000	2.000012	20.000024
24/06/2008	0.000000	2.000004	0.000000	20.000040	0.000004	2.000004	20.000038
26/06/2008	0.000000	2.000006	0.000002	20.000016	0.000000	2.000005	20.000015
04/07/2008	0.000000	2.000008	0.000008	20.000038	0.000010	2.000004	20.000029
10/07/2008	0.000000	2.000006	0.000000	20.000030	0.000014	2.000006	20.000023
11/07/2008	0.000000	2.000000	0.000004	20.000022	0.000000	1.999998	20.000020
15/07/2008	0.000000	1.999998	0.000000	20.000004	0.000000	1.999998	20.000004
17/07/2008	0.000000	2.000006	0.000000	20.000020	0.000014	2.000006	20.000013
18/07/2008	0.000000	1.999992	0.000000	19.999998	0.000000	1.999992	19.999998
21/07/2008	0.000000	1.999998	0.000000	20.000012	0.000000	1.999998	20.000012
23/07/2008	0.000000	2.000004	0.000000	20.000012	0.000000	2.000004	20.000012
24/07/2008	0.000000	2.000000	0.000000	20.000024	0.000002	2.000000	20.000023
29/07/2008	0.000000	1.999998	0.000002	19.999996	0.000000	1.999997	19.999995
05/08/2008	0.000000	1.999996	0.000000	19.999990	0.000000	1.999996	19.999990
06/08/2008	0.000000	2.000004	0.000012	20.000026	0.000014	1.999998	20.000013
07/08/2008	0.000000	2.000000	0.000000	20.000014	0.000000	2.000000	20.000014
19/08/2008	0.000000	1.999994	0.000000	20.000012	0.000000	1.999994	20.000012
28/08/2008	0.000000	2.000008	0.000002	20.000038	0.000020	2.000007	20.000027
03/09/2008	0.000000	2.000000	0.000008	20.000026	0.000012	1.999996	20.000016
09/09/2008	0.000000	2.000002	0.000000	20.000020	0.000006	2.000002	20.000017
11/09/2008	0.000000	2.000016	0.000010	20.000036	0.000014	2.000011	20.000024
16/09/2008	0.000000	2.000000	0.000000	20.000004	0.000000	2.000000	20.000004
17/09/2008	0.000000	2.000006	0.000008	20.000024	0.000000	2.000002	20.000020
18/09/2008	0.000000	2.000000	0.000000	20.000002	0.000000	2.000000	20.000002
22/09/2008	0.000000	2.000006	0.000000	20.000022	0.000000	2.000006	20.000022
23/09/2008	0.000000	2.000002	0.000000	20.000028	0.000000	2.000002	20.000028
24/09/2008	0.000000	2.000006	0.000010	20.000048	0.000016	2.000001	20.000035
29/09/2008	0.000000	2.000004	0.000000	20.000014	0.000000	2.000004	20.000014
30/09/2008	0.000008	2.000020	0.000018	20.000056	0.000040	2.000007	20.000027
01/10/2008	0.000000	2.000008	0.000000	20.000008	0.000008	2.000008	20.000004
02/10/2008	0.000000	2.000004	0.000000	20.000016	0.000010	2.000004	20.000011
07/10/2008	0.000000	2.000008	0.000000	20.000022	0.000000	2.000008	20.000022
15/10/2008	0.000000	2.000008	0.000010	20.000030	0.000018	2.000003	20.000016
16/10/2008	0.000000	1.999998	0.000006	20.000000	0.000018	1.999995	19.999988
21/10/2008	0.000000	2.000010	0.000012	20.000032	0.000014	2.000004	20.000019
22/10/2008	0.000000	2.000004	0.000000	20.000026	0.000000	2.000004	20.000026
23/10/2008	0.000000	2.000010	0.000000	20.000022	0.000012	2.000010	20.000016
29/10/2008	0.000000	1.999998	0.000000	20.000016	0.000000	1.999998	20.000016
30/10/2008	0.000000	2.000002	0.000000	20.000026	0.000000	2.000002	20.000026
03/11/2008	0.000000	2.000000	0.000000	20.000028	0.000000	2.000000	20.000028
04/11/2008	0.000000	2.000006	0.000004	20.000020	0.000016	2.000004	20.000010
05/11/2008	0.000000	2.000004	0.000002	20.000038	0.000016	2.000003	20.000029
11/11/2008	0.000000	2.000008	0.000000	20.000034	0.000008	2.000008	20.000030
12/11/2008	0.000000	2.000004	0.000000	20.000020	0.000000	2.000004	20.000020
14/11/2008	0.000000	2.000012	0.000006	20.000036	0.000014	2.000009	20.000026
17/11/2008	0.000000	2.000008	0.000000	20.000023	0.000006	2.000008	20.000020
18/11/2008	0.000000	2.000006	0.000002	20.000026	0.000002	2.000005	20.000024
19/11/2008	0.000000	2.000008	0.000000	20.000040	0.000000	2.000008	20.000040
24/11/2008	0.000000	2.000002	0.000000	20.000014	0.000000	2.000002	20.000014

5.4 Buoyancy correction guidance note

Good Practice Guidance *Note*

Buoyancy Correction and Air Density Measurement

Introduction

This Guidance Note gives recommendations of good mass measurement practice in calculating air density and buoyancy corrections but should not be considered a comprehensive guide.

Need for the Determination of Air Density

- Air is dense
 - A cubic metre of air weighs approximately 1.2 kilograms
 - A 1 kilogram stainless steel weight displaces 150 mg of air
- The density of air can vary between about 1.1 kg m⁻³ and 1.3 kg m⁻³, which is equivalent to a change of 25 mg in the weight of a stainless steel kilogram of volume 125 cm³.

What is the Typical Density of the Air?

- Standard air density is 1.2 kg m⁻³
 - This is the approximate density of air at 20 °C, 1013.25 mbar and 50% Relative Humidity
- For the range of density quoted in OIML R111 [1] (1.2 kg m⁻³ ± 10%)
 - Temperature is 20 °C ± 5 °C
 - Pressure is 1013.25 mbar ± 60 mbar
 - RH is 50% ± 30%
- For a temperature controlled lab the range of air densities experienced will be less (typically 1.2 kg m⁻³ ± 5%)

Determination of Air Density

- The usual method of determining air density is to measure temperature, pressure and humidity and calculate air density using the equation recommended by the Comité International des Poids et Mesures (CIPM) (derived by Giacomo [2] and modified by Davis [3]). The equation is not a perfect model of air density and introduces an uncertainty of approximately 1 part in 10⁴.
- Typical routine measurement capabilities for the air density parameters are indicated as follows with the best measurement capabilities in brackets:
 - Temperature to 0.1 °C (0.01 °C)
 - Pressure to 0.5 mbar (0.05 mbar)
 - Relative Humidity to 5% (0.25 °C dew point)
- The above parameters give a typical value on the air density (including the uncertainty from the equation) of 1 part in 10³ (2 parts in 10⁴ best capability)
- For lower accuracy measurements the NIST (simplified) air density formula can be used:

$$AD = \frac{[(0.348444 \times P) - h(0.00252t - 0.020582)]}{(273.15 + t)}$$

Where AD = Air density (kg m⁻³)
P = Air pressure (mbar)
h = Relative humidity of the air (%rh)
t = Air temperature (°C)

There is a typical uncertainty of 4 parts in 10⁴ on this equation over the range of air density of 1.2 kg m⁻³ ± 10%

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Druck Pressure Indicator



Protimeter Dew Point Meter



Edale model CD Thermometer

Figure 1: Typical Air Density Measurement Equipment

Air Density Artefacts

- The other common method for the determination of air density is to use artefacts of similar mass and surface area but different volumes.



Figure 2: Air Density Artefacts

- The difference between the apparent weight of the artefacts is proportional to the density of the air in which they are compared.

Variation in Buoyancy Effect with Varying Air Density

- In Standard Air of density 1.2 kg m^{-3}
 W – Conventional mass (kg)
 V – Volume (m^3)



If the conventional masses are equal ($W_1 = W_2$) the weights will balance in air of density 1.2 kg m^{-3}

- In Air of density 1.1 kg m^{-3}



As the air becomes less dense the larger weight (W_2) will appear heavier

Apparent weight difference
 $= (V_1 - V_2) \times (1.2 - 1.1) \text{ kg}$

- In Air of density 1.3 kg m^{-3}



As the air becomes more dense the larger weight (W_2) will appear lighter

Apparent weight difference
 $= (V_1 - V_2) \times (1.2 - 1.3) \text{ kg}$

When Should I make a Buoyancy Correction?

- Always be aware of the magnitude of the buoyancy effect and take it into account in your uncertainty budget

Material Compared with Conventional Stainless Steel	Effective Buoyancy Correction (ppm)
Platinum-Iridium	-94
Brass	-8
Stainless Steel*	7.5
Aluminium	294
Silicon	365
Water	1050

Table 1: Buoyancy correction when comparing materials of dissimilar density in air
*A correction of up to 7.5 ppm may be necessary when comparing stainless steel weights of different densities

- Air buoyancy is most significant when:
 - Performing high accuracy mass calibration (weights of OIML Class E₂ and better)
 - For weights whose density is not close to 8000 kg m⁻³ (except small fractional weights)
 - For measurements of other materials and liquids
 - For measurements in environments other than normal air (density 1.1 kg m⁻³ to 1.3 kg m⁻³)

How do I make a Buoyancy Correction?

Comparing weight W of volume V_W with standard S of volume V_S in air of density AD .

- True mass basis (TM)

$$T_W = T_S + (V_W - V_S) \times AD$$

- Conventional mass basis (CM)

$$C_W = C_S + (V_W - V_S) \times (AD - 1.2)$$

What do the OIML Specifications say?

- OIML R111 [1]
 - "The density of the material used for weights shall be such that a deviation of 10% from the specified air density (1.2 kg m⁻³) does not produce an error exceeding one quarter of the maximum permissible error."
- Thus:
 - OIML tolerance for weights > 100 grams of class E₂ is 7810 kg m⁻³ to 8210 kg m⁻³
 - Maximum error is: $T_S \times \left(\frac{1}{7810} - \frac{1}{8000} \right) \times (1.32 - 1.2)$
 - Or 0.365 mg on 1 kg (maximum allowable error 1.5 mg)
- But:
 - The normal uncertainty quoted on an E2 kilogram is 0.5 mg therefore the buoyancy error is significant
 - The buoyancy error is however, less significant in an air density range of 1.18 to 1.22 for a controlled laboratory environment.

Conventional and True Mass

■ True mass

- A measure of the amount of substance of an artefact
- The constant of proportionality between F and g ($F=mg$)
- Measured by comparison between the masses of artefacts (and defined by the mass of the international prototype kilogram)

■ Conventional Mass

- For a weight taken at 20 °C, the conventional value of mass is the mass of a reference weight of a density of 8000 kg m⁻³ which it balances in air of density 1.2 kg m⁻³ (OIML Recommendation R33 [4])

- How they are related

$$M_T = M_C + (V_{8000} - V_{\gamma}) \times 1.2$$

■ When should they be used?

- Conventional mass

- For all OIML class weights (generally)
- For standard weights (or artefacts) whose density is close to 8000 kg m⁻³

- True Mass

- For the highest accuracy mass calibration
- For weights whose density is not close to 8000 kg m⁻³ (except small fractional weights)
- For measurements of other materials and liquids
- For measurements in environments other than normal air (density 1.1 kg m⁻³ to 1.3 kg m⁻³)



Figure 3: 1 kg of Stainless Steel compared with 1 kg of platinum-iridium

Related Good Practice Guides

- Guide to the measurement of mass and weight
- Guide to the measurement of pressure and vacuum
- Guide to the measurement of force
- Good Practice Guide on Cleaning, Handling and Storage of Weights
- Good Practice Guidance note on Thermal Effects on Balances and Weights

References

- [1] International Organisation of Legal Metrology (OIML). International Recommendation No 111: 1994. Weights of classes E1, E2, F1, F2, M1, M2, M3.
- [2] Giacomo P. "Equation for the density of moist air". Metrologia, 1982, 18, 33-40.
- [3] Davis RS. "Equation for the density of moist air". Metrologia, 1992, 29, 67-70.
- [4] International Organisation of Legal Metrology (OIML). International Recommendation No 33: 1979. Conventional value of the result of weighing in air.

For further information contact:

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www.npl.co.uk/npl/md/

5.5 Certificate of calibration for temperature, pressure and humidity logger



REPORT NUMBER: CAL-08-03-13-005
 DATE OF CALIBRATION: 03/13/2008
 NEXT CALIBRATION DUE: 03/13/2009
 PAGE NUMBER: 1 OF 3

N.I.S.T CALIBRATION CERTIFICATE

CALIBRATION LABORATORY:
 CALIBRATION REQUIRED:
 APPLICABLE DOCUMENTS:

OMEGA Electrical Calibration Laboratory
 OM-CP-PRHTemp2000-122
 OM-CP-PRHTemp2000 Calibration Procedure
 OM-CP-PRHTemp2000 Calibration Log
 Humidity Systems Report No. 2008.22014
 TTI, Inc. Certificate Number P1158

Software Calibration:

A convenient feature of the OM-CP-PRHTemp2000 is that it can be calibrated through software. This means that no manual adjustments are necessary for the OM-CP-PRHTemp2000 to achieve its rated accuracy. All adjustments are entered through our software and stored in non-volatile memory within the device. Once these adjustments are made, the data is automatically corrected during the download process.

Temperature Calibration:

To determine the temperature calibration value for the OM-CP-PRHTemp2000 it is necessary to compare the actual value read by an uncalibrated OM-CP-PRHTemp2000 to the value that should be read by the device. The difference is called the calibration value. An example would be if the OM-CP-PRHTemp2000 read 23.5°C when it should have read 23.0°C, then the user would enter 0.5°C for the calibration value. The 0.5°C is then automatically subtracted from all readings during the download and the readings are calibrated. This is called a single-point calibration. OMEGA verifies the temperature readings at room temperature using a Rotronic hygrometer Model number IC3 with a temperature accuracy of $\pm 0.3^\circ\text{C}$ at 23.0°C. The serial number of this device is 47197004/52993003. This calibration is NIST traceable per report # 2008.22014.

Calibration Environment

Temperature: 25.0 °C

Before Calibration:

Serial Number	Calibration Temperature (°C)	Temperature Read (°C)	Temperature Offset Correction (°C)
M88683	25.0	25.2	0.0

After Calibration:

Serial Number	Calibration Temperature (°C)	Temperature Read (°C)	Temperature Offset Correction (°C)
M88683	25.0	25.0	0.2

Calibration Statement:

OMEGA Engineering, Inc. certifies that the above instrumentation has been calibrated and tested to meet or exceed the published specifications for temperature measurement. This procedure was performed using instrumentation and standards that are traceable to the National Institute of Standards and Technology. This certificate shall not be reproduced, except in full, without the written consent of OMEGA Engineering, Inc.



REPORT NUMBER: CAL-08-03-13-005
 DATE OF CALIBRATION: 03/13/2008
 NEXT CALIBRATION DUE: 03/13/2009
 PAGE NUMBER: 2 OF 3

Humidity Calibration:

To determine the humidity calibration values for the OM-CP-PRHTemp2000 it is necessary to compare the actual value read by an uncalibrated OM-CP-PRHTemp2000 at known humidity levels to the values that should be read by the device. The OM-CP-PRHTemp2000 uses an internal humidity sensor. OMEGA calibrates each device in an environmental chamber. The humidity levels produced by this chamber at equilibrium are measured using a Rotronic HygroClip IC-3 Thermo-Hygrometer, serial number 47197004/52993003. This device is specified by Rotronic to have an accuracy of ± 1 %RH at $25^{\circ}\text{C} \pm 5^{\circ}\text{C}$. The reference sensor is calibrated by Humidity Systems and is NIST traceable per report # 2008.22014.

<u>Calibration Environment 1</u>		<u>Calibration Environment 2</u>	
Temperature:	25.0 °C	Temperature:	25.0 °C
Relative Humidity:	26.0 %RH	Relative Humidity:	75.0 %RH

Before Calibration:

Serial Number	Humidity Read in Environment 1 (%RH)	Humidity Read in Environment 2 (%RH)	Humidity Gain Correction	Humidity Offset Correction (%RH)
M88683	27.4	76.4	1.0	0.0

After Calibration:

Serial Number	Humidity Read in Environment 1 (%RH)	Humidity Read in Environment 2 (%RH)	Humidity Gain Correction	Humidity Offset Correction (%RH)
M88683	27.4	76.4	1.0	0.0

Calibration Statement:

OMEGA Engineering, Inc. certifies that the above instrumentation has been calibrated and tested to meet or exceed the published specifications for relative humidity measurement. This procedure was performed using instrumentation and standards that are traceable to the National Institute of Standards and Technology. This certificate shall not be reproduced, except in full, without the written consent of OMEGA Engineering, Inc.



REPORT NUMBER: CAL-08-03-13-005
 DATE OF CALIBRATION: 03/13/2008
 NEXT CALIBRATION DUE: 03/13/2009
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Pressure Calibration:

To determine the pressure calibration values for the OM-CP-PRHTemp2000 it is necessary to compare the actual value read by an uncalibrated OM-CP-PRHTemp2000 at two different pressure levels to the values that should be read by the device. Since two points must be used, this is called a two-point calibration. In this case, the OM-CP-PRHTemp2000 uses an internal pressure sensor, which is supplied by Honeywell/SenSym ICT. OMEGA verifies the pressure readings at with an SI Pressure Instruments PC6 Pro Pressure Calibrator model number PC6-B-5A-40-C, serial number 5909 with accuracy of ± 0.025 %FS at 21°C . Tracable certificate number is P1158. This calibration is NIST traceable.

<u>Calibration Environment 1</u>		<u>Calibration Environment 2</u>	
Temperature:	25.0 °C	Temperature:	25.0 °C
Pressure Input:	14.485 PSIA	Pressure Input:	29.00 PSIA

Before Calibration:

Serial Number	Pressure Read in Environment 1 (PSIA)	Pressure Read in Environment 2 (PSIA)	Pressure Gain Correction	Pressure Offset Correction (PSIA)
M88683	14.62	29.104	1.000	0.0

After Calibration:

Serial Number	Pressure Read in Environment 1 (PSIA)	Pressure Read in Environment 2 (PSIA)	Pressure Gain Correction	Pressure Offset Correction (PSIA)
M88683	14.482	29.00	0.99779552	0.16792946

Calibration Statement:

OMEGA Engineering, Inc. certifies that the above instrumentation has been calibrated and tested to meet or exceed the published specifications for temperature measurement. This procedure was performed using instrumentation and standards that are traceable to the National Institute of Standards and Technology. This certificate shall not be reproduced, except in full, without the written consent of OMEGA Engineering, Inc.

Authorized signature: _____

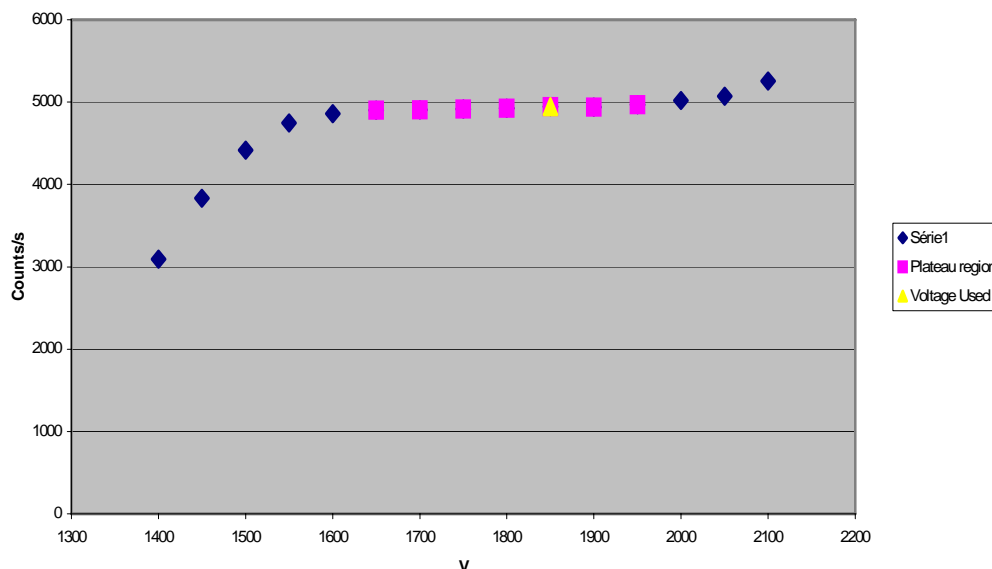
Shimma

Date: _____

3/13/2008

5.6 Beta counter plateau

The voltage applied to the proportional counter was altered and the beta count rate (corrected for background and dead-time effects) was monitored, as shown below. The operating voltage was chosen as 1850 V. Double click on the picture to access the numbers.

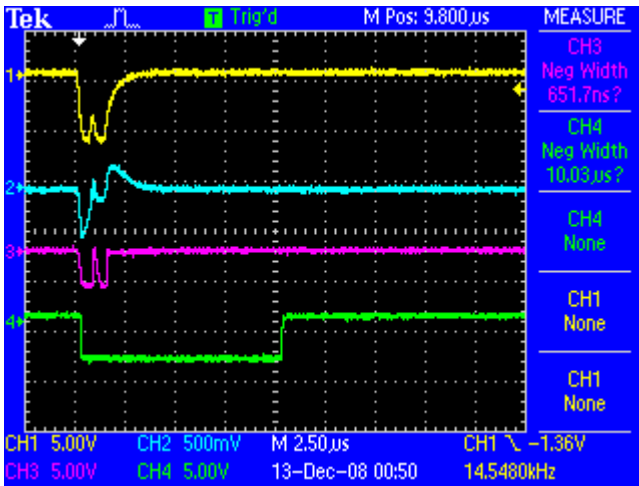


5.7 Beta channel deadtime measurement

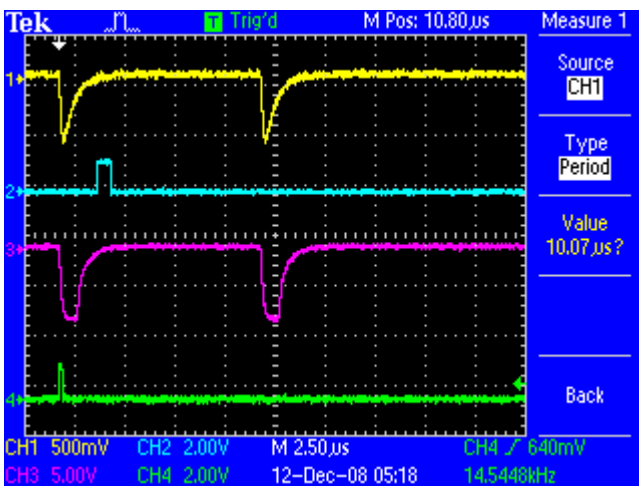
The NPL “in-house” beta channel amplifier unit used combines the functions of signal amplification of input pulses from the pre-amplifier, imposition of a discriminator level (on a differentiated signal) and imposition of a non-extendable dead time of desired length.

The behaviour of the unit (particularly in relation to the effects of “pulse pile-up”), is best described by considering the response to test pulses supplied by a dual-pulser unit of regular frequency, with variable delay between the first and second pulses from each “event”.

The dual-pulser “delay” was set so that the amplified dual pulses pile-up (as shown in the yellow trace for CH1 below). The beta channel amplifier unit differentiates the amplified signals (as shown in the blue trace for CH2 below) and the discriminator operates on this differentiated signal. The minimum resolvable pulse-separation in this instance was around 0.65 μs (as shown in the pink trace for CH3 below) although the input signals had a width of around 3 μs . These logic pulses following the imposition of the discriminator are used as the input to the non-extendable dead time of around 10 μs duration (as shown in the green trace for CH4 below).

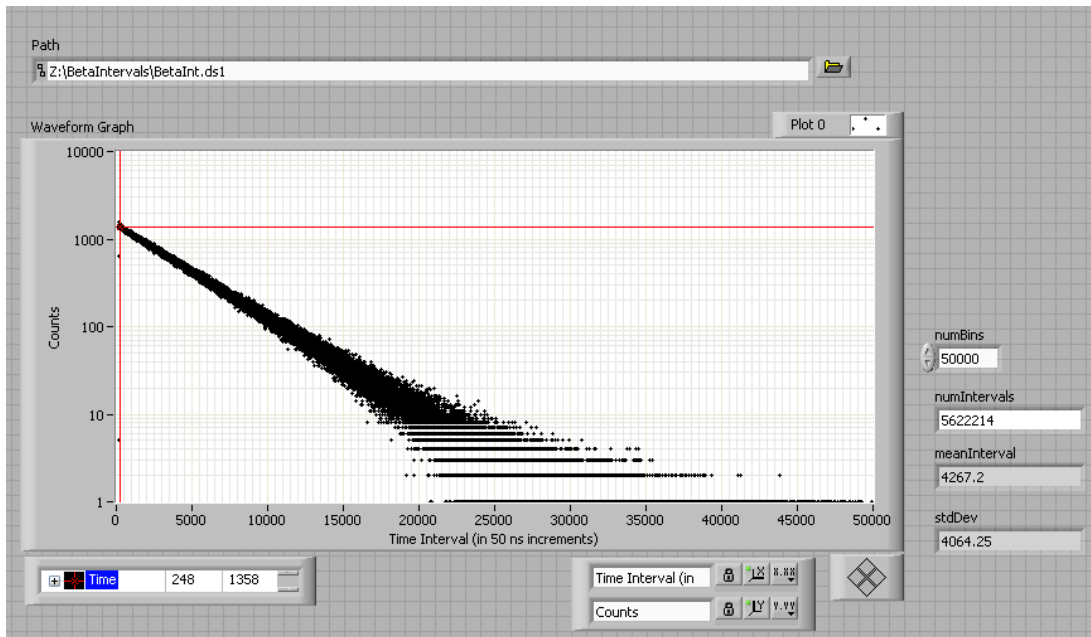


However, the width of the logic pulses for CH4 are taken from a “test point” within the amplifier unit itself, and additional circuitry providing the logic pulses for subsequent counting imposes some further dead time. The measurement of the imposed dead time is best performed by monitoring these logic pulses as the dual-pulser delay is varied, until the second of the shaped pulses just vanishes, as shown below:



In this instance, the yellow trace (CH1) shows the pulses from the dual-pulser unit (used as the input to the beta channel amplifier) and the blue trace (CH2) shows a trigger signal from the dual pulser. The pink trace (CH3) shows the amplified pulses and the green trace (CH4) represents the output from the discriminator/dead time circuitry. The dual-pulser delay was adjusted so that the second output on CH4 just disappears (and thus not shown). The dead time was measured as 10.07 μ s.

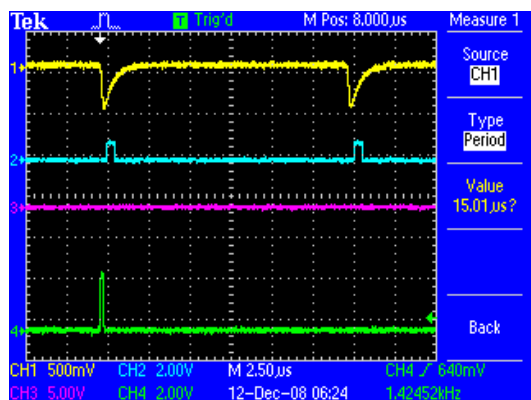
As a further check on the dead time from the beta channel, the interval distributions from a real Co-60 source were collected using the NPL Digital Coincidence Counting (DCC) system, with the results shown below:



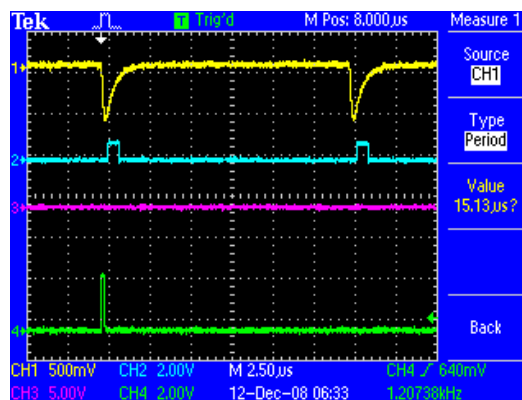
The Mean Interval from the beta channel was measured as 4267.2 ADC clock “ticks”, where the ADC clock frequency was 20 MHz. This represents a mean interval of 213.36 μ s. The standard deviation of the interval distribution was measured as 4064.25 clock “ticks” (or 203.21 μ s). If one assumes that the original Poisson Process is perturbed solely by the imposition of a dead time of the non-extendable type, then the difference between the mean and standard deviation yields an estimate of the imposed dead time of 10.15 μ s. The beta channel non-extendable dead time was estimated as 10.1(1) μ s.

5.8 Gamma channel deadtime measurement

A similar procedure (based on the use of a dual pulser) for dead time measurement as described in the previous section was used to determine the dead times for each of the gamma gates, yielding estimates for $\tau_{\gamma 1}$ and $\tau_{\gamma 2}$ of 15.0 μs and 15.1 μs respectively.



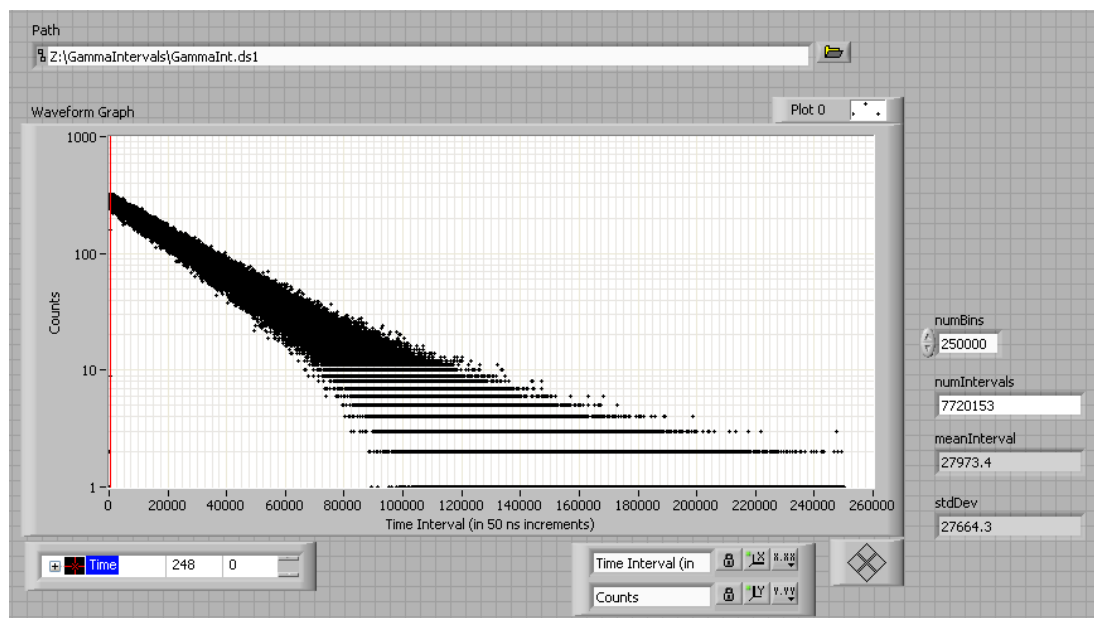
Gamma Gate 1



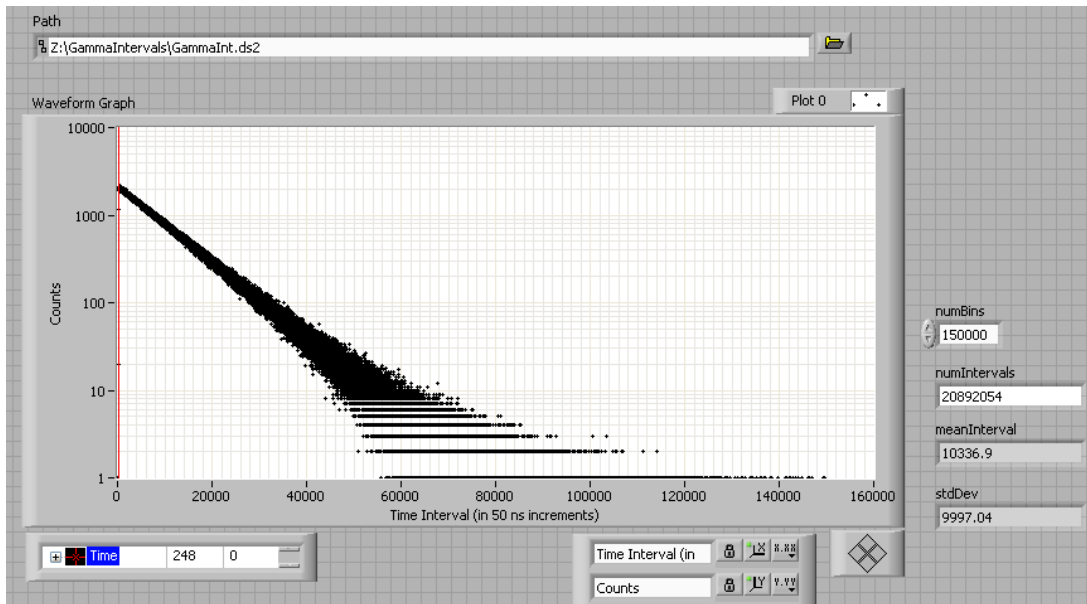
Gamma Gate 2

However, the SCAs employed in the gamma channels operate on the summed pulses from the shaping amplifiers, and not on a differentiated signal (as was the case for the beta channel amplifier). Thus, the effects of pulse pile-up are not minimised.

Consider the Gamma Channel interval-distributions for each gamma gate:



Gate 1: Mean Interval: 27973.ADC clock ticks; Std.Dev of intervals: 27664.3. The difference between the mean and the standard deviation yields an estimate of the gamma dead time of 15.4 μs .



Gate 2: Mean Interval: 10336.9 ADC clock ticks; Std.Dev of intervals: 9997.04. The difference between the mean and the standard deviation yields an estimate of the gamma dead time of 17.0 μ s. The minimum observed interval was 300 ADC clock ticks (or 15 μ s).

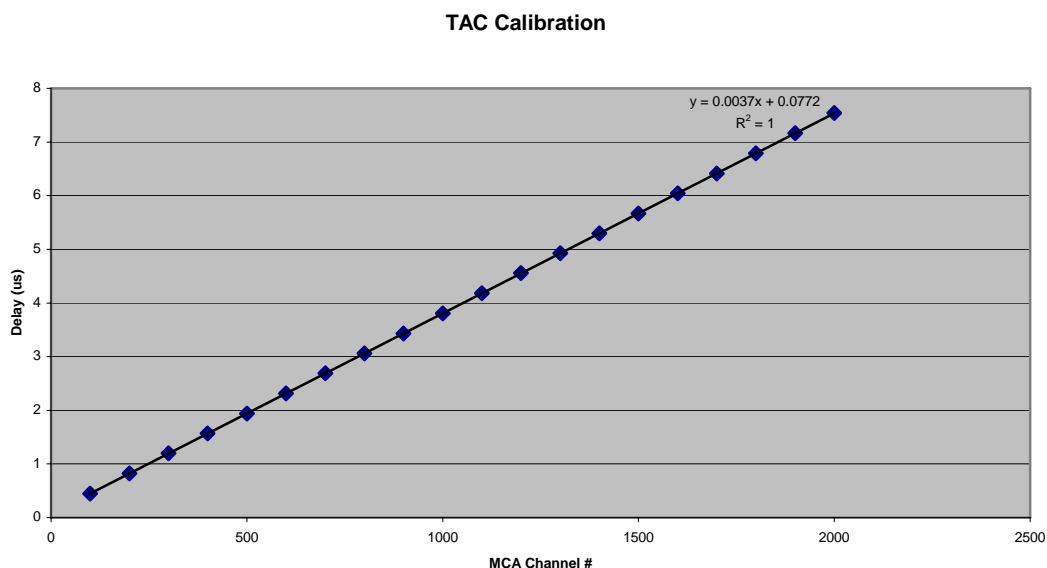
These estimates differ somewhat from the previously estimated dead times, as the concept of using the difference between the mean and standard deviation of the interval distribution as an estimate of the imposed non-extendable dead time relies on a Poisson Process forming the input pulse stream. The effects of pulses pile-up are evident, and the interval distribution has deviated from the expected “shifted exponential” shape associated with a pure Poisson Process perturbed by a non-extendable dead time.

The gamma channel non-extendable dead times were estimated as 15.0(5) μ s and 15.1(5) μ s respectively.

5.9 Coincidence channel resolving time measurement

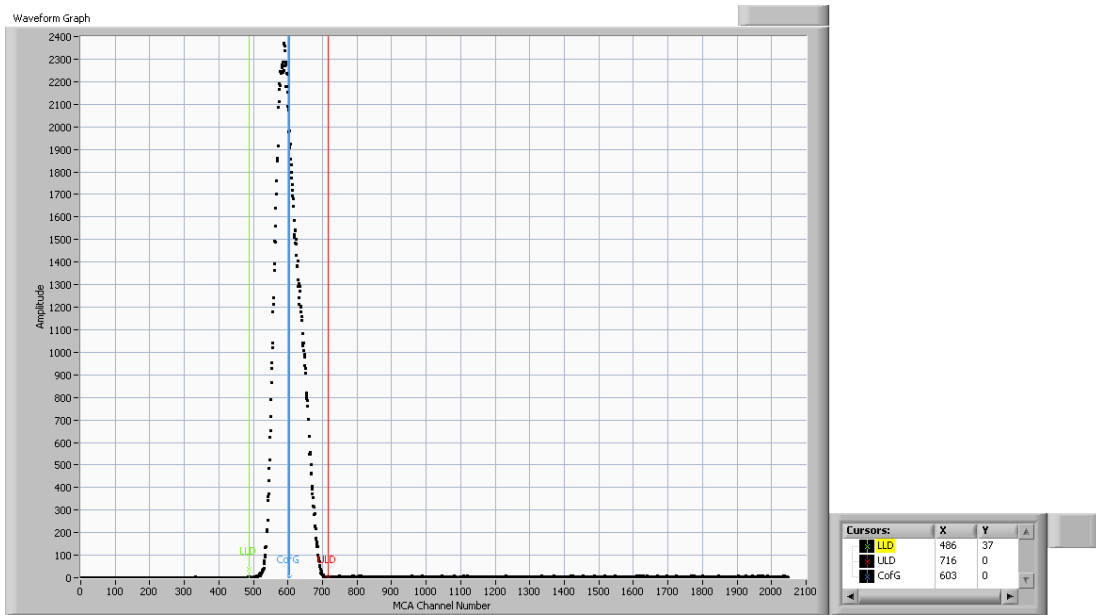
5.9.1.1 Calibration of TAC

A dual-pulsar unit was employed as the input to a Time to Amplitude Converter (TAC), with the output TAC pulses sent to an MCA. The delay employed was measured on a calibrated oscilloscope as well as a calibrated Philips PM6680 counter timer unit.

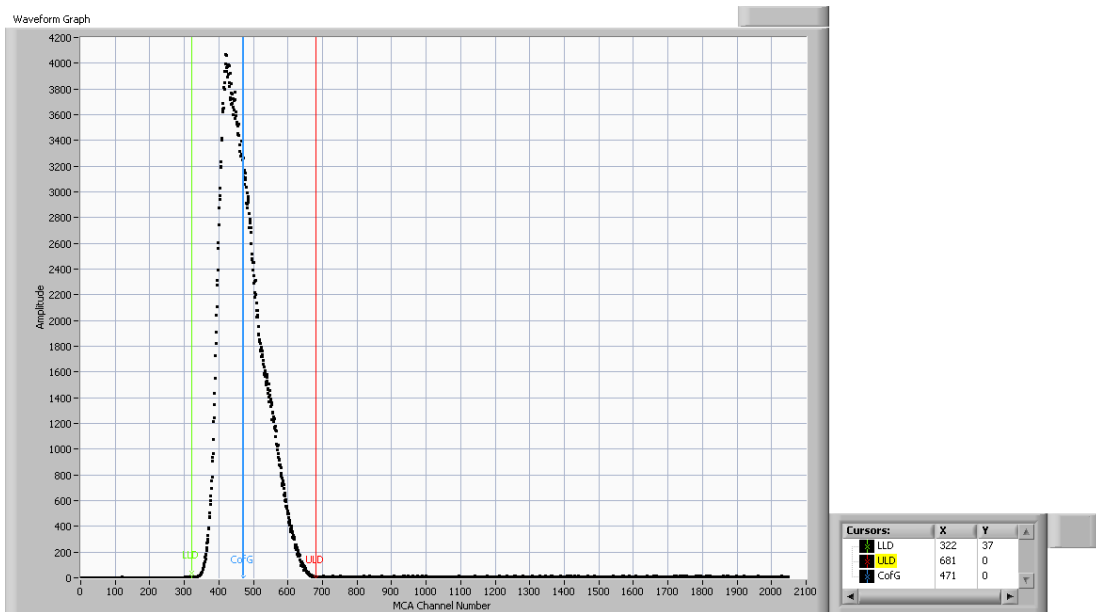


MCA Channel #	Delay (scope) μs	Delay (PM6680) μs
100	0.448	0.448
200	0.824	0.823
300	1.197	1.195
400	1.57	1.57
500	1.941	1.94
600	2.315	2.314
700	2.689	2.688
800	3.063	3.062
900	3.432	3.432
1000	3.805	3.805
1100	4.18	4.179
1200	4.555	4.557
1300	4.925	4.924
1400	5.298	5.297
1500	5.67	5.669
1600	6.044	6.042
1700	6.415	6.414
1800	6.788	6.787
1900	7.164	7.163
2000	7.54	7.534
	slope	0.00373

5.9.1.2 *beta-gamma delay distributions and coincidence resolving times*



Gate 1: Range of time jitter = 0.86 μ s



Gate 2: Range of time jitter: 1.34 μ s

The timing of the pulses were adjusted so that there was zero mean delay between the arrival of “coincident” beta and gamma events, and the coincidence resolving times were set to just fully encompass the time-delay distributions. These were measured on the calibrated oscilloscope as:

$$r_{\beta 1} = 0.50(1) \mu\text{s}, \quad r_{\beta 2} = 0.70(1) \mu\text{s}$$

$$r_{\gamma 1} = 0.50(1) \mu\text{s}, \quad r_{\gamma 2} = 0.70(1) \mu\text{s}$$

5.9.1.3 Confirmation of Coincidence Resolving Times

The real pulses were replaced by two (independent, unrelated) regular pulsers, and the number of counts in the beta, gamma and both coincidence channels were monitored over a series of 10 second intervals.

Reading #	Nb	Ng	Nc1	Nc2
1	145481	163868	2387	3329
2	145482	163863	2364	3336
3	145483	163862	2401	3329
4	145482	163861	2385	3330
5	145483	163861	2354	3307
6	145485	163860	2379	3333
7	145482	163855	2374	3307
8	145482	163852	2383	3354
9	145482	163848	2388	3336
10	145482	163848	2376	3331
mean	145482.4	163857.8	2379.1	3329.2
s.d.	1.074968	6.729701991	13.18627	13.7905

The sum of the resolving times is then simply $Nc/(Nb Ng)*10$, yielding:

$$(r_{\beta 1} + r_{\gamma 1}) = 0.998 \mu s$$

and

$$(r_{\beta 2} + r_{\gamma 2}) = 1.397 \mu s$$

which is in excellent agreement with the previously measured widths of the logic pulses comprising the respective resolving times.

5.10 Sample output from BGC program

For each source measurement, the BGC program outputs data in the following format:

```

expects mmt 1 identifier s08080 wt=15.269300 (or a background)

 960798 145771 130897 394224 356227 @ 1229382104 Mo Dec 15 15:01:44 08
data files= c:\bgc\350_old c:\bgc\350

mmt 1 identifier s08080
      wt= 15.269-for 5 cycles hl=1925.2d reftime= 2008y 12m 1d 12h
      b/eb 1/eb-1 b 1-eb eb eg
g1 cycle 1 354.85 0.06803 332.24 0.06370 0.9363 0.13652
g2 cycle 1 354.94 0.06832 332.24 0.06395 0.9360 0.37422
(campion 354.75)
959533 145353 130776 393952 356131 @ 1229382309 Mo Dec 15 15:05:09 08
g1 cycle 2 353.68 0.06600 331.78 0.06192 0.9381 0.13657
g2 cycle 2 354.32 0.06792 331.78 0.06360 0.9364 0.37461
(campion 354.13)
958024 145772 131160 393138 355481 @ 1229382514 Mo Dec 15 15:08:34 08
g1 cycle 3 353.11 0.06605 331.23 0.06196 0.9380 0.13719
g2 cycle 3 353.66 0.06769 331.23 0.06340 0.9366 0.37450
(campion 353.47)
958771 145604 130943 393464 355644 @ 1229382719 Mo Dec 15 15:11:59 08
g1 cycle 4 353.56 0.06654 331.51 0.06239 0.9376 0.13686
g2 cycle 4 354.07 0.06807 331.51 0.06373 0.9363 0.37439
(campion 353.88)
960546 146506 131734 395151 357042 @ 1229382924 Mo Dec 15 15:15:24 08
g1 cycle 5 354.29 0.06666 332.15 0.06249 0.9375 0.13743
g2 cycle 5 354.89 0.06846 332.15 0.06407 0.9359 0.37519
(campion 354.70)

NewAuto
summary mmt 1 identifier s08080 wt= 15.269 ( 5x200secs):
      g1 means 353.90 0.06665 331.78 0.06249 0.9375 0.13691
      obs pc sd 0.085 0.550 0.057 0.515 0.034 0.129
      th pc sd 0.057 0.685 0.046 0.642 0.043 0.115

      g2 means 354.37 0.06809 331.78 0.06375 0.9362 0.37458
      obs pc sd 0.069 0.201 0.057 0.188 0.013 0.044
      th pc sd 0.048 0.374 0.046 0.350 0.024 0.059
  
```

NB: The bracketed term “(campion xxx.xx)” may be ignored, and is only displayed when the calculated value for “b/eb” using the Cox-Isham-Smith high-order approximation for the estimation of the coincidence channel rate (ICRU 52 Equations 5.51, 5.55 – 5.57) differs from that calculated using the Campion formula by more than 0.03%.

5.11 Masses used as divisor of countrates

The masses used as the divisor of the countrates were those calculated from the substitution method:

Source ID	Mass (mg)
s08078	15.8076
s08079	14.8802
s08080	15.2693
s08081	15.1627
s08082	15.8665
s08083	16.102
s08084	15.6784
s08085	33.9465
s08086	32.8687
s08087	31.7576