



Comparison of Activity Concentration Measurement of ^{63}Ni and ^{55}Fe in the Framework of the EUROMET 297 Project

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Eleven laboratories participated in an intercomparison of activity concentration measurements using liquid scintillation counting (LSC) for the standardization of ^{63}Ni and ^{55}Fe in the frame of the EUROMET project No. 297 and under the co-ordination of LPRI. The purpose of this action was to compare the main LSC activity concentration measurement methods currently used in radioactive metrology, and to exchange models and ideas on LSC. This paper presents a summary of the results reported by the participant laboratories and an overview of the measurement methods used. © 1998 The National Physical Laboratory. Elsevier Science Ltd. All rights reserved

Introduction

In May 1993, nine laboratories agreed to participate in a comparison of activity concentration measurements for the liquid scintillation counting (LSC) standardization of ^{63}Ni and ^{55}Fe in the frame of the EUROMET project No. 297. The purpose of this action was the comparison of the main LSC activity concentration measurement methods, and the exchange of models and ideas on LSC.

The two main LSC methods used in radioactivity metrology are the CIEMAT/NIST efficiency-tracing method and the triple to double coincidence ratio (TDCR) method. The CIEMAT/NIST method can use any kind of LS counter but requires the use of standard counting source of some other radionuclide like tritium. The TDCR method does not need a reference standard, but requires a specific 3-photomultiplier-tube (PMT) counter. The detection efficiency algorithms used in these methods are mainly based on the same physical models but these models have been developed in an independent way; therefore they can slightly differ on some

points and the evaluation of sample quenching is not the same.

The aim of the EUROMET action was also to take this opportunity to standardize two important radionuclides, ^{63}Ni and ^{55}Fe , which were selected for the following reasons:

— ^{63}Ni is a pure- β emitter with a 66 keV maximum energy. LSC is a good method for calibrating the radioactivity concentration of such a radionuclide, and this calibration is of practical interest as this product is a tracer for the nuclear power industry.

— ^{55}Fe decays by electron capture and emits low energy (i.e. about 5 keV) electrons and X-rays. The calibration of this nuclide is of practical interest and cannot be easily done by other means.

The results of this EUROMET project are described in a report (Cassette, 1996) and the main points are summarized in this article.

Participants

Nine laboratories initially agreed to participate in this action: BIPM, CIEMAT, ENEA, IRMM, LPRI, NAC, NIST, PTB, RC. During the

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ICRM'95 meeting in Paris, two new laboratories were asked to join this action: *CMI* and *SCK-CEN*.

Radioactive Solutions

The action started in 1994 with ^{63}Ni but the measurements were inconclusive, mostly because it was observed by all participants that the solution distributed by LPRI gave unstable LS sources in Ultima Gold[®] scintillator. It was then decided to discard the results. In November 1995, LPRI sent to each participant an ampoule of ^{63}Ni solution containing $36\ \mu\text{g g}^{-1}$ of Ni^{++} in 1 M HCl and an ampoule of ^{55}Fe solution containing $2.8\ \mu\text{g g}^{-1}$ of Fe^{3+} in 1 M HCl.

Equipment

The measurements were made by each participating laboratory using various LSC equipments, mostly commercial instruments for the application of the CIEMAT/NIST method and locally-developed counters for the application of the TDCR method.

Measurement of ^{63}Ni

Each participant laboratory prepared its own set of LSC sources with the ^{63}Ni solution and their own scintillator cocktail. The scintillator cocktails used were: Ultima Gold[®], Ready Safe[®], Supersolve[®], Insta-Gel[®], PCS[®] and Hisafe[®].

The sources prepared with Ultima Gold[®] were reported to be stable by CIEMAT, ENEA, IRMM, LPRI and RC. NIST recommends the addition of blank water to the counting source to obtain an aqueous fraction greater of at least 2% to 3% in order to achieve tractable tracing against the tritium standards. However no special problem (bias or instability) was found at LPRI with Ultima Gold[®] sources using the ^{63}Ni EUROMET solution, irrespective of the water content of the source. BIPM reported the instability of LSC ^{63}Ni sources made with Beckman ReadySafe[®] LS cocktail.

Calculation of the detection efficiency

For the implementation of the CIEMAT/NIST method, it seems that each laboratory has developed its own version of the original programs EFFY from CIEMAT (Garcia-Toraño and Grau Malonda, 1985), but no major modifications of the initial calculation models were made. The laboratories used various ^3H standards which are not directly relatable to each other.

The laboratories using the TDCR method have developed their own software for the calculation of the detection efficiency. The programs used by NAC are based on the algorithms of the EFFY program. The programs used by LPRI and RC are based on the same models, and were recently homogenized.

Activity concentration measurement results

The results reported by the participant laboratories are presented in Fig. 1 and Table 1.

The CIEMAT/NIST method was used by 7 laboratories with various ^3H standards. The agreement between these measurement is rather good, the relative standard deviation being 0.7%. Three laboratories used the TDCR method but, in this case, the relative standard deviation is slightly higher, *ca.* 1%. CMI used an extrapolation method, the activity concentration being calculated from the relation between the coincidence count rate and a function of the ratio of the coincident count rate and the sum count rate. The SCK-CEN value was obtained by an LSC relative method using a ^{63}Ni LMRI commercial standard. Inasmuch as the LMRI standard is traceable to the ^{63}Ni LPRI standard, the SCK-CEN value is strongly correlated to the LPRI value (Table 2).

The dispersion between the activity concentration of the ^{63}Ni solution measured by the 11 laboratories is quite low (*ca.* 1.4%). In order to get a realistic evaluation of the mean value of the activity concentration of the solution, it must be observed that:

— the SCK-CEN value was not measured with an absolute method and is strongly correlated to the LPRI value,

Table 1. Activity concentration of the ^{63}Ni EUROMET 297 solution

Laboratory	Activity concentration (kBq g^{-1})	Standard uncertainty (kBq g^{-1})	Relative standard uncertainty (%)
BIPM (CIEMAT/NIST)	39.9	0.1	0.25
CIEMAT (CIEMAT/NIST)	39.96	0.17	0.43
CMI	41.77	2.13	5.10
ENEA (CIEMAT/NIST)	40.28	0.22	0.55
IRMM (CIEMAT/NIST)	40.1	0.2	0.50
LPRI (TDCR)	39.95	0.21	0.53
NAC (TDCR)	39.59	0.2	0.51
NAC (CIEMAT/NIST)	39.52	0.17	0.43
NIST (CIEMAT/NIST)	39.8	0.16	0.40
PTB (CIEMAT/NIST)	40.22	0.34	0.85
RC (TDCR)	40.34	0.27	0.67
SCK-CEN	40	1	2.50
Mean	40.12	0.58 (standard deviation)	1.44
Weighted mean	39.9	0.71 (intraclass std. dev.)	1.78

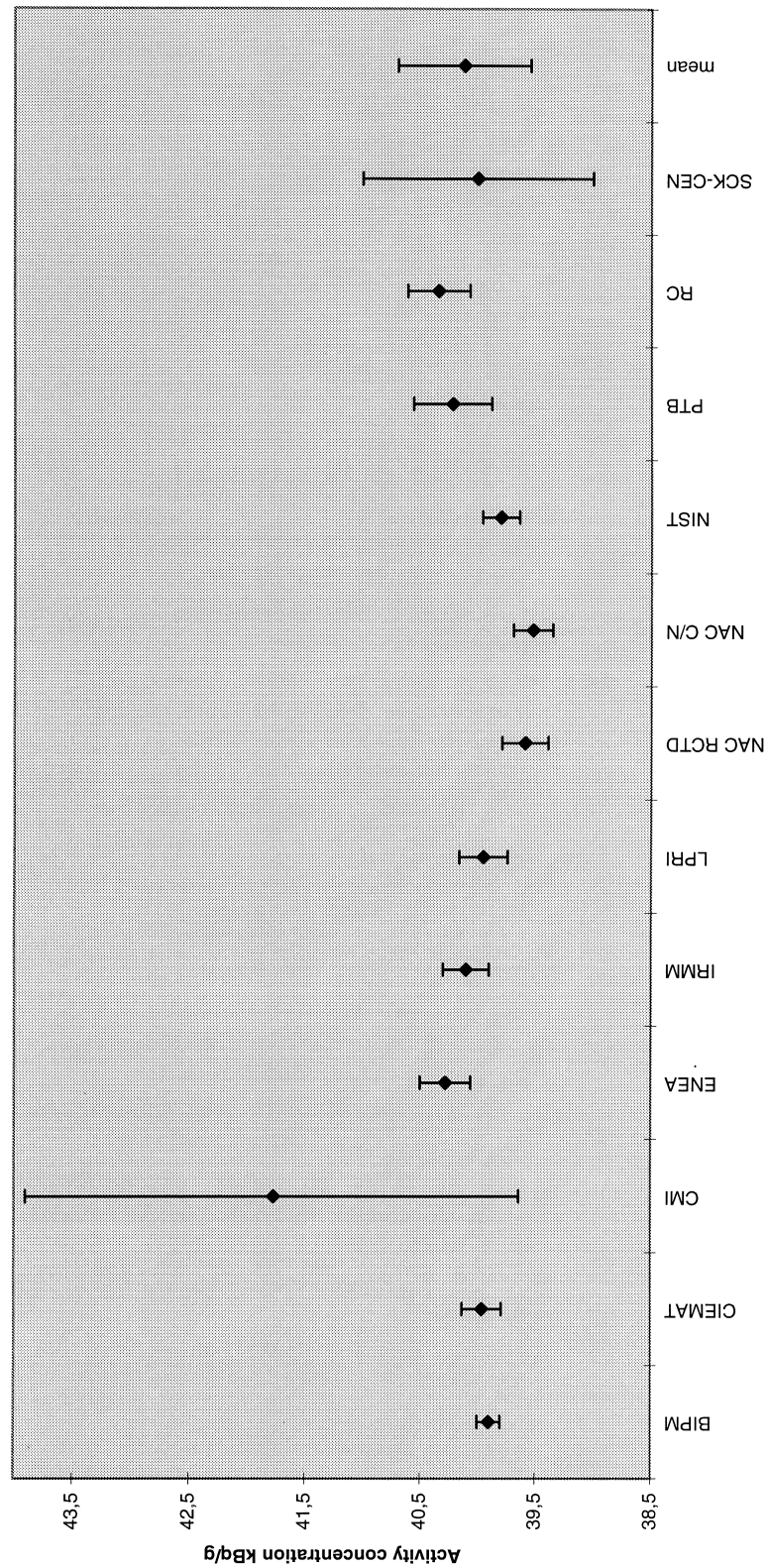


Fig. 1. Activity concentration of the ^{63}Ni EUROMET 297 solution.

— the measurement value given by CMI value is not compatible with those given by the other participants both in terms of its centered value and its reported uncertainty.

If these two values are excluded, the mean of the 10 remaining values is 39.97 kBq g^{-1} with a standard uncertainty of 0.26 kBq g^{-1} (0.7% relative). All the 10 values are compatible within one standard deviation, and their associated standard uncertainties are all of the same order of magnitude.

RC made some recent changes in the detection efficiency calculation program and informed the coordinator that the activity concentration value would change to $39.92 \pm 0.26 \text{ kBq g}^{-1}$ (Broda, 1997).

Some laboratories made additional comments which are reported hereafter.

NIST observed that the ^{63}Ni $E_{\beta-\text{max}}$ value ($65.87 \pm 0.2 \text{ keV}$) and associated half-life value ($100.1 \pm 2.0 \text{ a}$) which was recommended by the coordinator, are obsolete considering recent publications. A best estimate of these values would be ($66.945 \pm 0.004 \text{ keV}$) for $E_{\beta-\text{max}}$ (ENSDF data file) and ($101.1 \pm 1.4 \text{ a}$) for the half-life. NIST used these latter values and observed that the use of the recommended values would have increased the mass activity of the solution by an average of *ca.* 0.40 to 0.45%. The recommended ^{63}Ni $E_{\beta-\text{max}}$ value was used by all the other participants. LPRI observed that this change in the $E_{\beta-\text{max}}$ would have a negligible effect on the activity result obtained using the TDCR method. NIST also pointed out that, for the application of the CIEMAT/NIST method, the chemical composition of the ^3H sources and of the ^{63}Ni sources must be very well matched in order to obtain very accurate activity measurement results.

IRMM reported that the use of Canberra Packard ^3H standards resulted in a similar ^{63}Ni detection efficiency curve to the one obtained with a ^3H LPRI standard up to tSIE quench indicating

parameter of *ca.* 350. Above that value, the Canberra Packard standards exhibited gradually higher efficiencies with relative discrepancies of *ca.* 2% for a tSIE value of 600. The overall effect of these discrepancies would be an apparent activity concentration of the ^{63}Ni solution that is 0.4% lower than the value reported by IRMM. NIST used both the ^3H LPRI standard and a ^3H NIST standard. Differences between the CIEMAT/NIST tracing method using these two standards are *ca.* 0.18%, systematically. The other laboratories using the CIEMAT/NIST method used various ^3H standards. As the traceability of these standards is not known, the influence of this factor on the results is unpredictable and can be considered only to have a random influence on the results.

RC and LPRI used an extrapolation method to determine the proper value of the ionization quenching parameter, the kB constant of the Birks formula. This determination is done by decreasing the detection efficiency of the LS counter and finding out the value of the kB constant which will give an activity concentration that is independent of the detection efficiency. At RC, efficiency modification is done by defocusing the PMT; at LPRI, it is achieved by adding coaxial grey filters around the LSC vials. The best estimate obtained at RC is $0.0107 \text{ cm MeV}^{-1}$. LPRI observed a dependence of the kB factor on the chemical composition of the LS source, which ranged from $0.011 \pm 0.002 \text{ cm MeV}^{-1}$ for 50 mg of ^{63}Ni solution in 10 ml of Ultima Gold[®] to $0.006 \pm 0.002 \text{ cm MeV}^{-1}$ for the same sources with the addition of 100 mg of 1 M HCl.

NAC observed a correlation between the activity results and the choice of the scintillator with both the CIEMAT/NIST and the TDCR methods. In the NAC experimental conditions, the mean activity concentration of the Insta-Gel[®] sources are *ca.* 0.7% higher than the one obtained with Ultima Gold[®] sources using the TDCR method. The

Table 2. Activity concentration of the ^{55}Fe EUROMET 297 solution

Laboratory	Activity concentration (kBq g^{-1})	Standard uncertainty (kBq g^{-1})	Measurement method
BIPM	47.4	0.4	CIEMAT/NIST
CIEMAT Insta-Gel	53.09	1	CIEMAT/NIST
CIEMAT Ultima Gold	52.68	1	CIEMAT/NIST
CMI	50.46	2.52	LSC
ENEA	50.6	0.6	CIEMAT/NIST
IRMM Ultima Gold	50.2	0.4	CIEMAT/NIST
IRMM Insta-Gel	53.1	0.3	CIEMAT/NIST
LPRI	50.5	0.2	tracer ^{54}Mn
LPRI	50.4	2.3	Si detector
LPRI	50.4	1.1	Prop. countet
LPRI	51	0.2	TDCR
NAC	52.71	0.42	tracer ^{54}Mn
NAC	49.16	0.4	CIEMAT/NIST
NAC	48.7	0.4	TDCR
NIST	52.95	1.18	Relative method
PTB	51.1	0.35	CIEMAT/NIST
RC	51.2	0.47	TDCR

results of Insta-Gel[®] sources are 0.4% lower than for the Ultima Gold[®] sources using the CIEMAT/NIST method. In both cases, the kB value used is $0.0075 \text{ cm MeV}^{-1}$. CIEMAT used the same kB value.

IRMM reported that the efficiency tracing method using the Packard Instruments protocol gave a significantly 2.3% higher value of the activity concentration of the ^{63}Ni solution than the CIEMAT/NIST method.

Additionally, it is interesting to note that NIST observed that the re-assay of NIST (SRM 4226) ^{63}Ni standard, performed at the same time of this action, agreed with the present NIST value to $+0.095\%$ using the ^3H LPRI standard and -0.081% using the ^3H NIST standard. The various ^{63}Ni standardizations done at NIST over a period of 27 years have been summarized by Collé and Zimmerman (Collé and Zimmerman, 1996).

Conclusions

Deployment of the CIEMAT/NIST and the TDCR methods resulted in very similar values for the activity concentration of the ^{63}Ni solution. This conclusion is remarkable if one considers that these measurements were made using various LS counters and various ^3H standards. As this radionuclide has a quite low maximum beta energy, it can be considered to be a good test of the relative compatibility of these two measurement methods for pure beta emitters. Although evaluations of the respective uncertainty components differ noticeably from one laboratory to another, the reported values are of similar magnitude for both methods.

The main uncertainty contributor for the CIEMAT/NIST method is the uncertainty in the activity of the ^3H standard. For the TDCR method, the main source of uncertainty is the ionization quenching factor.

The effect of the particular choice of the $E_{\beta - \max}$ value on the detection efficiency seems to be lower for the TDCR method than for the CIEMAT/NIST method.

This action has also shown that special attention must be paid to the carrier concentration of the ^{63}Ni solution in order to obtain tractable LSC sources in Ultima Gold[®] LS cocktail and that the quality of the measurements depends strongly on the stability of the sources.

Measurement Of ^{55}Fe

Each participant laboratory prepared its own set of LSC sources using the ^{55}Fe solution and used its own scintillation cocktail. The stability of the LS sources was studied by the participants and the reported results are somewhat strange. Ultima Gold[®] LSC sources containing a few tens of mL of the EUROMET ^{55}Fe solution were found to be stable by BIPM, ENEA, LPRI and RC. With the same cocktail, however, CIEMAT observed an instability (-1% over 10 d). The stability was restored by the addition of $50 \mu\text{L}$ of a carrier solution ($100 \mu\text{g g}^{-1}$ of Fe^{3+} in 1 M HCl) in 10 mL of LS cocktail. IRMM also found that their sources were unstable with an apparent activity decay of 0.2% per day. The origin of these differences in the behavior of Ultima Gold[®] cocktail under nearly identical conditions is not apparent. A possible explanation might be cocktail ageing effects or differences between batches of the commercially prepared scintillation fluids.

Insta Gel[®] was used by CIEMAT, IRMM, and NAC and the sources made with this cocktail were reported to be stable.

BIPM observed instability of sources prepared with Readysafe[®].

LPRI and RC used a home-made cocktail containing butyl-PBD (2 (4'-*t*-butylphenyl)-5-(4"-biphenyl)-1,2,3,4-oxadiazole) and bis-MSB (*p*. bis-(*o*-methylstyryl)-benzene) in toluene. For the use of that scintillator, the ^{55}Fe solution was complexed with TOPO (trioctylphosphinoxid) dissolved in ethanol before the incorporation of the cocktail. The sources prepared by this method were found to be stable and gave reproducible activity measurement results.

Scintillator and ^{55}Fe data

The detection efficiency of ^{55}Fe is, *a priori*, very dependent on the nuclear and atomic data of this radionuclide. The critical parameters are P_K , (the K-electron capture probability), the Auger electron energies and, to a lesser extent, ω_K , the KX fluorescence yield. The data used by the laboratories are in good agreement, except for ω_L , but the influence of this parameter on the calculation of the detection efficiency is very low, because the absorption probability of the L-shell X-rays in the scintillator is almost 1. The value of P_K used by most partici-

Table 3. X-ray absorption probability in LS-cocktail as evaluated by nine laboratories for three X-ray emissions of ^{55}Fe

	CIEMAT UG	CIEMAT IG	ENEA UG	NAC IG	PTB UG	RC UG	RC Toluene	LPRI UG	RV UG
X_{KL}	0.9427	0.9423	0.949 ± 0.008	$0.927 \text{ (mean } K_L, K_M)$	0.963	0.947	0.930	0.96 ± 0.01	0.949 ± 0.008
X_{KM}	0.9240	0.9230	0.932 ± 0.008		0.9486	0.929	0.907	0.94 ± 0.01	0.932 ± 0.008
X_{LM}	0.9998	0.9998	1	1	1	1	1	1	1

UG: Ultima Gold, IG: Instagel, RV: recommended values.

pants is 0.881, PTB using a slightly different value of 0.8848.

The detection efficiency is also dependent on the probability of absorption of X-rays in the LS cocktail. A set of recommended values was proposed by the co-ordinator for Ultima Gold[®] cocktail, but other values were calculated by some participants, as shown in Table 3. These values were calculated with stochastic models using evaluated or calculated values of μ_x , the cocktail linear absorption coefficient. PTB calculated μ_x for Ultima Gold[®] with the XCOM program (Berger and Hubbell, 1987) using an atomic composition of the cocktail deduced from a chemical analysis (i.e. C: 0.791, H: 0.096, O: 0.097, P: 0.016). The values of μ_x for Ultima Gold[®] were measured at LPRI using a ⁵⁵Fe source, a thin absorption cell and a germanium detector (Plagnard and Cassette, 1996). The probabilities of absorption of K-shell X-rays in Ultima Gold[®] calculated by PTB and LPRI are in good agreement but *ca.* 2% higher than the values calculated by CIEMAT, ENEA and RC.

Detection efficiency calculation

The CIEMAT/NIST method was used initially by BIPM, CIEMAT, ENEA, IRMM, NAC, NIST and PTB. Three laboratories, NIST, ENEA and PTB, observed the failure of the method for the standardization of ⁵⁵Fe using a ³H standard. These laboratories explained this problem by incompatible definitions of the free parameter in the program EFFY, used for the ³H standard, and in the program EMI used for electron capture nuclides. For that reason, NIST did not use the CIEMAT/NIST method for the measurement of ⁵⁵Fe, ENEA developed its own software (MINERVA) for the calculation of the detection efficiency, and PTB used ⁵⁴Mn instead of ³H as a standard. NAC reported that the CIEMAT/NIST method gave 6 to 7.5% lower activity concentration results than the ⁵⁴Mn tracer method. No problems on the method were reported by BIPM, CIEMAT, and IRMM.

The TDCR method was used by LPRI, NAC and RC. These laboratories used locally developed programs for the calculation of the detection efficiency of ⁵⁵Fe. NAC observed that, like the CIEMAT/NIST method, the TDCR method gave an activity concentration of the ⁵⁵Fe solution *ca.* 7.6% lower than the activity concentration measured using a ⁵⁴Mn tracing method.

CMI used an extrapolation measurement method in LSC. The activity concentration is calculated from the relation between the coincidence counting rate and the ratio of the coincidence to the summing counting rate of the phototubes, the efficiency being changed by changing the discrimination levels. CMI also made measurements with a pressurized proportional counter calibrated with ⁵⁴Mn, ⁵⁷Co, ⁶⁵Zn and ⁵¹Cr.

NAC used a $4\pi(e,X)-\gamma$ coincidence method using ⁵⁴Mn to trace the efficiency. Sources were counted with ⁵⁴Mn alone, followed by counting of the mixed ⁵⁴Mn + ⁵⁵Fe, and no extrapolation was used. The efficiency calculations were based on the measured ⁵⁴Mn efficiency in the mixed source.

LPRI used a $4\pi(e,X)-\gamma$ coincidence method with ⁵⁴Mn to trace the efficiency. The electron detector was a proportional counter and the γ -ray detector a 3" × 3" NaI(Tl) scintillator. LPRI also used two semiconductor detectors for the activity measurement of ⁵⁵Fe by X-ray spectrometry: a HP-Ge detector with a beryllium window, and a Si(Li) detector with a beryllium window calibrated with a monoenergetic synchrotron beam. LPRI also used a dedicated defined solid angle proportional counter for the measurement of ⁵⁵Fe X-ray emission (Plagnard, 1996).

NIST assay of the activity concentration of the ⁵⁵Fe solution was based on comparative measurements against a ⁵⁵Fe NIST standard (SRM 4929E). The comparison was made using either QIP-adjusted quench corrections or the CIEMAT/NIST method efficiency tracing against the ⁵⁵Fe NIST standard with the EMI code.

Activity measurement results

The results reported by the participants are presented in Fig. 2. For their final ⁵⁵Fe activity concentration results, BIPM, CIEMAT, ENEA and IRMM used the CIEMAT/NIST method with a ³H standard, and PTB used the CIEMAT/NIST method with a ⁵⁴Mn standard. LPRI and RC used the TDCR method. CMI used a LSC extrapolation method, NAC used a $4\pi(e,X)-\gamma$ tracer method with ⁵⁴Mn, and NIST used a relative method against their own ⁵⁵Fe standard.

The mean activity concentration value is 51.05 kBq g⁻¹ with a standard uncertainty of 1.65 kBq g⁻¹. The relative standard uncertainty is *ca.* 3%.

Discussion

The dispersion between the activity concentration of the ⁵⁵Fe solution measured by 10 laboratories is quite high (*ca.* 3%). The results can be divided in two groups and an isolated result. The first group of 6 results (CMI, ENEA, IRMM, LPRI, PTB and RC) has a mean value of 50.8 kBq g⁻¹ and an associated standard deviation of 0.4 kBq g⁻¹ (0.8% relative). The second group of 3 results (CIEMAT, NAC and NIST) has a mean value of 52.85 kBq g⁻¹ with a standard deviation of 0.13 kBq g⁻¹ (0.2% relative). The isolated result (BIPM) is 47.4 kBq g⁻¹. Some observations can be made from all the results reported by the participants, including those from non LSC measurements.

There is no obvious correlation between the results and the employed measurement methods. Nevertheless, in the case of the CIEMAT/NIST

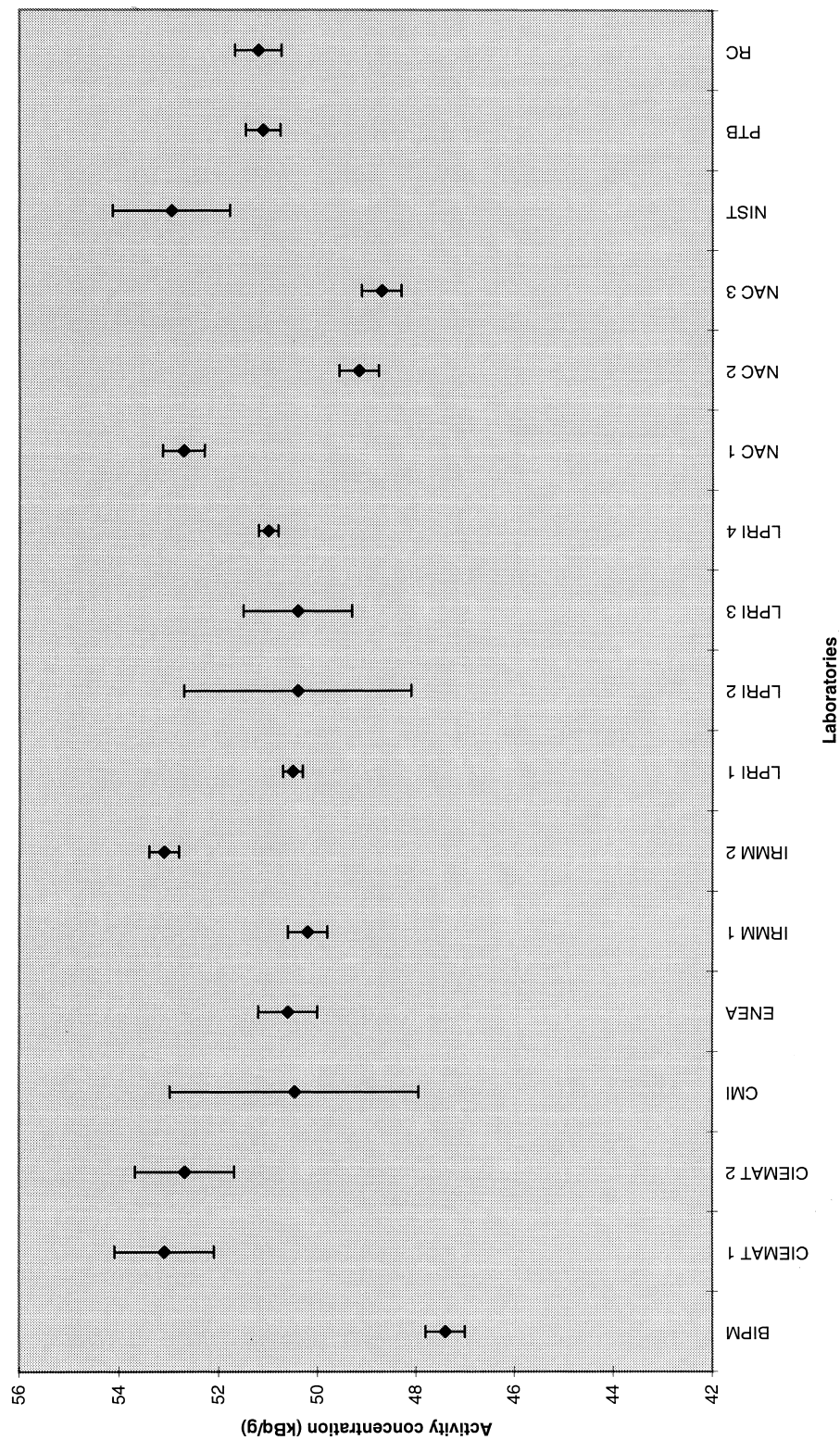


Fig. 2. Activity concentration of the ⁵⁵Fe EUROMET 297 solution.

method, as the efficiency calculation computer codes vary from one laboratory to another, it would be imprudent to say that these laboratories really used the same measurement method. For the TDCR method, the number of laboratories using this method is not large enough to draw any statistically pertinent conclusion.

It seems that there is a correlation between the results and the scintillator cocktail used. CIEMAT observed a small systematic difference between the activity concentration results obtained with Ultima Gold[®] and InstaGel[®] cocktail. This effect was also observed by IRMM but in a much more dramatic extend; there is more than a 5% difference between the results obtained with these two scintillator cocktails. No discrepancies were observed by RC with Ultima Gold[®] and a toluene-based home-made cocktail.

NAC reported that the TDCR or CIEMAT/NIST results are 6 to 7.6% lower than the ⁵⁴Mn tracer method, and NAC has not yet an explanation for this discrepancy. LPRI also observed a small systematic (*ca.* 1%) discrepancy between the TDCR and the ⁵⁴Mn tracer method results.

There is no obvious correlation between the measurement results and the ⁵⁵Fe nuclear and atomic data used.

Some versions of CIEMAT/NIST programs EFFY and EMI seem to be incompatible for the implementation of the CIEMAT/NIST method to measure activity concentrations of ⁵⁵Fe with a ³H standard. This was observed by ENEA, NIST and PTB. ENEA reported that the use of EFFY and EMI programs gave a ⁵⁵Fe activity concentration result of $66.97 \pm 1.63 \text{ kBq g}^{-1}$, a value more than 30% higher than the one obtained with the ENEA code, MINERVA!

The stability of Ultima Gold[®] cocktail used by many laboratories in this comparison is puzzling

and needs further study. The influence of this finding on the dispersion of the results is unknown.

Conclusions

The standardization of the EUROMET 297 ⁵⁵Fe solution raises many questions concerning the stability of the LSC sources, the implementation of the CIEMAT/NIST method and systematic bias versus ⁵⁴Mn tracing methods. The discrepancy in the results by several percent shows that the standardization of this nuclide is still an open problem.

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