

DISCUSSION

RECOVERY FROM TRACER CONTAMINATION IN AMS SAMPLE PREPARATION

A J T JULL, D J DONAHUE and L J TOOLIN
NSF Accelerator Facility for Radioisotope Analysis
University of Arizona, Tucson, Arizona 85721

Samples that contain high levels of tracer ^{14}C are occasionally received at radiocarbon laboratories. Enriched samples of about 10 times modern can usually be handled without severe degradation of sample blanks, due to sample memory effects. It is not possible to identify such samples in advance, and one should be more wary of certain sources of ^{14}C samples than others. We report here on a recent experience in our laboratory, similar to that reported by Vogel *et al* above.

In May 1989, we processed two samples of CO_2 gas through our normal gas-handling system, which uses a known volume and a capacitance manometer. The samples were then reduced to graphite as usual (Slota *et al* 1987). The first sample was measured to be ca 200 times modern, and the second was ca 5000 times modern. These samples were not run for more than two minutes on our accelerator. The samples were immediately removed from the accelerator, and blank samples, fabricated prior to receipt of the "hot" samples, were loaded. These samples confirmed that after some minutes of sputtering with Cs, that the accelerator blank was at its usual value. Subsequent to these "hot" samples, we processed a blank CaCO_3 sample through our sample preparation procedure. This sample was measured to contain ^{14}C at a level of $82.6 \pm 0.6\%$ that of modern carbon. After this result, the gas handling line was flushed with dead CO_2 and also degassed by heating the glassware. Two subsequent blanks measured one and three days later gave 7.6% and 1.4% modern, respectively. These samples were made into graphite on lines other than the one (no. 7) which had been used for the "hot" samples. After these measurements, we endeavored to identify and eliminate any potential source of "tracer" ^{14}C which was the cause of the elevated blank. The results of these measurements are given in Table 1. All the blank measurements listed were performed using commercial CO_2 , which we normally find to have ca 0.4% modern carbon (Linick *et al* 1986). From the results presented in the table one can conclude the following:

1. Cross-contamination between a hot sample and a blank sample fabricated in the same glass vacuum system is of the order of 0.016%, so that processing a blank sample immediately after an oxalic-II sample would result in a blank of ca 0.02% modern.
2. Flushing of the system with "dead" CO_2 reduces the contamination from the tracer levels to ca 1.4%. Subsequent re-equilibration of the sample handling system with "dead" CO_2 did not improve this.
3. Subsequent tests of blanks showed variable ^{14}C , which we were eventually able to ascribe to contamination of some gas storage vessels. Test 4 involved replacement of the Kel-F plug in the Kontes high-vacuum valves in the gas storage vessels, and this did not eliminate the contamination.
4. The contaminated graphite line could not be cleaned up, as shown by test 5.

CONCLUSIONS

After these investigations, we concluded that the only way to ensure low and repeatable blanks was to replace the entire gas-handling system, including an MKS Baratron gauge, the contaminated graphite line (no. 7), including the pressure transducer in the graphite line, and all gas sample vessels in the laboratory. When these materials were removed from the laboratory and replaced, normal blanks of 0.4% or better were again achieved. This experience can only serve to reinforce our belief that tracer ^{14}C should not be allowed in a

TABLE 1
Results of blank and test measurements subsequent to processing of a tracer ^{14}C sample

Target	Date run	Description	Graphite line	Per cent modern
1. <i>Tracer samples</i>				
4607	7 May	CO ₂ gas sample	7	20,000%
4611	8 May	CO ₂ gas sample	7	500,000%
2. <i>Blank tests subsequent to tracer samples. Gas measured in contaminated volume.</i>				
4613	9 May	blank 1 day after tracer	1	82.6%
4617	11 May	blank 3 days after tracer	1	7.6%
4625	14 May	blank 5 days after tracer	8	1.4%
3. <i>Test combustion line after equilibration with CO₂ for 3 days</i>				
4627 A	15 May		1	1.9%
B	15 May		4	4.7%
4. <i>Test of gas sample storage vessels</i>				
4629	16 May	Gas vessel stored in air for 5 days, then baked out, and pumped in vacuum oven. O-rings on stopcock replaced.	4	1.3%
4630	16 May	Same as 4629, O-rings not replaced	5	1.3%
5. <i>Replacement of stopcock plug and O-rings on contaminated graphite line</i>				
4632	16 May	Tank CO ₂	7	6.4%

radiocarbon laboratory. In fact, from the data presented in the table, we would expect that a sample containing 20 times the ^{14}C of a modern sample would temporarily double our contamination background.

As additional illustration of the dangers of tracer ^{14}C in a low-background laboratory, we note that in our laboratory we measured many blank targets prepared at the Carnegie Institution in Washington. The room in question at Carnegie was used as a tracer ^{14}C laboratory in the 1960s (Stafford, pers commun). Despite vigorous recent efforts to clean up the room, the “blanks” we measured all had ^{14}C contents equivalent to modern or even post-bomb levels. These cleaning efforts were eventually abandoned.

REFERENCES

- Linick, TW, Jull AJT, Donahue, DJ and Toolin, LJ 1986 Operation of the NSF-Arizona Accelerator Facility for Radioisotope Analysis and results of selected collaborative research projects. *In* Stuiver, M and Kra, RS, eds, Internat ^{14}C conf, 12th, Proc. *Radiocarbon* 28 (2A):522–533.
- Slota, PJ, Jull, AJT, Linick, TW and Toolin, LJ 1987 Preparation of small samples for ^{14}C accelerator targets by catalytic reduction of CO. *Radiocarbon* 29 (2):303–306.
- Vogel, JS, Southon, JR and Nelson, DE 1989 Memory effects in an AMS system: catastrophe and recovery. *Radiocarbon*, this issue.