REG/CR-0289

MEASUREMENT AND STANDARDS FOR NUCLEAR MATERIAL SAFEGUARDS

Quarterly Report
February 1 - April 30, 1978

National Bureau of Standards

Prepared for
U. S. Nuclear Regulatory Commission

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NUREG/CR-0289
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MEASUREMENTS AND STANDARDS FOR NUCLEAR MATERIALS SAFEGUARDS

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February 1 - April 30, 1978

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Manuscript Completed: July 1978
Date Published: August 1978

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U. S. Department of Commerce
Washington, D.C. 20234

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Office of Nuclear Regulatory Research
U. S. Nuclear Regulatory Commission
Under Contract No. AT(49-25)-9009
NRC FIN No. B-6016

PREFACE

This report was prepared for the U.S. Nuclear Regulatory Commission, Office of Nuclear Regulatory Research under Interagency Agreement No. AT(49-25)-9009.

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ABSTRACT

This report is a review of the third quarter, (February 1, 1978 thru April 30, 1978), of a long-term NBS program sponsored by NRC to up-grade national measurements and standards capability for nuclear materials safeguards.

The overall approach that NBS is utilizing to provide for development and dissemination of a consistent set of national measurement standards for nuclear materials safeguards is presented. It should be stressed that a great deal of work needs to be done to provide the standardization base for alternate fuel cycles. Many materials such as thorium, Uranium 233, and plutonium or mixed oxides "spiked" with fission products might well be found in future alternate fuel cycles. The NBS program is aimed at providing both the standards for today's needs and the standards for future fuel cycles.

A summary of the progress for each of the five tasks in the project is given.

OVERVIEW OF NBS PROGRAM

An adequate measurements and accounting system is necessary for the detection of and protection against surreptitious removal of special nuclear material by persons having authorized access to facilities. The sensitivity of this type of detection depends directly on the uncertainties of measurement. The NBS program will assure the availability of the standards, reference measurement methods, and quality assurance methodology for the adequate control of measurements for safeguards. Domestic and international dissemination is required.

The goal of the proposed NBS program is:

> To assure that timely measurement of special nuclear material in both today's fuel cycles and in future alternate fuel cycles, can be performed at reasonable cost with accuracy sufficient for the safeguarding of nuclear material. These measurements of enriched uranium, plutonium, and related materials need to be made by both inspectors and the industry.

The NBS program in measurements and standards for safeguarding of nuclear materials consists of three related parts: (1) calibration standards, reference measurement methods, sampling schemes, statistical treatment of data and data generation; (2) dissemination mechanisms to transfer the standards and reference methods and data to the users; and (3) mechanisms to directly assist inspectors and the nuclear industry to ensure that their measurements are of sufficient accuracy.

In order to carry out the tasks assigned to NBS in a timely manner, NBS must continually assess the advancing needs for national measurement standards. NBS must also provide the broad technical base that is needed to carry out the program. NBS has received substantial guidance and input from NRC as to standards needs. Continued input from all appropriate NRC offices (NRR, NSS, SD, IE) will be extremely helpful and are solicited.

NBS is using a multidisciplinary, matrix management approach to carry out the program. Under the new NBS reorganization, work is being carried out in six line organizations: Center for Radiation Research, Center for Thermodynamics and Molecular Science, Center for Analytical Chemistry, Center for Materials Science, Center for Applied Mathematics and the Center for Mechanical Engineering and Process Technology. Researchers with backgrounds in analytical chemistry, mass and volume, nuclear and radiation physics, thermodynamics, mechanics, etc. are part of the program team. NBS is also supplying a substantial amount of equipment, both old and new, that is needed to carry out the program.

PROGRESS OF TASKS

Task I: DEVELOPMENT AND IMPLEMENTATION OF MEASUREMENT ASSURANCE PROGRAMS AND STATISTICAL SUPPORT FOR NUCLEAR MATERIALS SAFEGUARDS

Principal effort this quarter was devoted to the accountability tank volume calibration effort. Several statistical techniques are under investigation: the jackknife, nonparametric model fitting, and Scheffe's approach among others. Programs were written, and others are being obtained elsewhere, to compare these methods. Preliminary results were presented to two groups at NBS concerned with volume calibration. Investigation of possible implementations of the Measurement Assurance Program (MAP) concept in volume and liquid flow measurements will begin next quarter. Also a new computer program for spline fitting will be obtained and tried out on actual in-plant volume calibration data.

Dr. H. H. Ku, NBS Statistical Engineering Division Chief, was appointed to the SALE Steering Committee. He will participate in the meeting of that committee, and another statistician will participate in the general SALE meeting in May. Following these meetings, efforts will be started toward utilizing the SALE machinery to expedite the measurement assurance approach in these measurement areas.

Miscellaneous Items: Reviewed three papers for Nuclear Materials Management and a Nuclear Regulatory Commission (NRC) draft report, collaborated on analysis of data for certification of SRM 950b, U_3O_8 for uranium assay, arranged for one statistician to spend half time for one to three months with the Mass Spectrometry group at NBS in order to become familiar in depth with the procedures used so that we might better model the measurement process, participation at several meetings, interacted with flow calibration and UF_6 MAP effort, participated in program review at NRC, assisted with ASTM Committee C-26 work, and were visited by A. Maimoni of Lawrence Livermore Laboratory (LLL). This visit led to a small effort by Operations Research personnel of the NBS Center for Applied Mathematics, investigating the applicability of their skills to the validation of models used to evaluate the effectiveness of safeguards systems.

Personnel: C. Spiegelman joined the Statistical Engineering Division (SED) and the safeguards effort on March 20, 1978. Dr. Spiegelman was an assistant professor at the University of Florida and a member of a consulting firm. Several other candidates were interviewed for the vacancies remaining.

Projections for next quarter, not mentioned previously: Continued C-26 committee activity, including finishing the draft PuO_2 powder sampling document before the C-26 meeting in July; attendance at the Institute for Nuclear Materials Management (INMM) meetings, including the working groups INMM 8.1, 8.2, and 9.4; presentation to INMM 8.2 on the nonparametric calibration work now going on at NBS.

Investigators for this task are J. Lechner, C. Spiegelman, H. Ku and C. Reeve.

Task II: STANDARDIZATION OF DESTRUCTIVE ANALYTICAL CHEMISTRY METHODOLOGY FOR NUCLEAR MATERIALS

A. Measurement Assurance Programs

1. Alpha Spectrometry-76

Evaluation of the National Bureau of Standards and Euratom's Central Bureau for Nuclear Measurements (CBNM)'s data for the isotopic characterization of four plutonium samples for the Alpha Spectrometry-76 interlaboratory test has been completed. The values determined by thermal ionization mass spectrometry were used as the baseline to which all counting experiments were compared. A close examination of the mass spectrometric data (Table 1) indicated a systematic difference for the ^{238}Pu composition of three samples. The most apparent causes are residual ^{238}U which was not removed by chemical purification, ^{238}U from the filament material, or an organic background peak. Present efforts to determine the source of the differences may require reanalysis of these samples as well as other materials. Even with the small discrepancy in the ^{238}Pu measurements, it was found that the agreement between the two laboratories was satisfactory in all cases, and especially in light of a preliminary evaluation of the alpha spectrometry data of the participating laboratories. These results indicate large and unacceptable levels of uncertainty by some of the participants in measuring Pu isotopic composition.

2. Safeguards Analytical Laboratory Evaluation (SALE)

NBS and New Brunswick Laboratory (NBL) are jointly characterizing SALE materials to determine the "true" value for use in the SALE program. Samples of depleted SALE uranium metal were received from NBL for both isotopic and chemical assay characterization. Both the SALE uranium metal and NBS SRM 960, a high purity metal of known uranium content, have been reduced to the appropriate size for assay by coulometry

and the modified NBL Davies-Grey titrimetric method. At present, samples of SRM 960 are being analyzed by coulometry and the NBL method prior to analysis of the SALE uranium for which there is a limited amount of material.

B. Standard Reference Materials

1. Natural Uranium Isotopic SRM

The grinding and blending of a pitchblende ore which is to be certified as a natural uranium isotopic SRM was completed. The original bulk pitchblende, consisting of several large rocks totaling 5 kg, was reduced to 4.9 kg of 200 mesh material. The preliminary objective of pulverizing the rocks to a size which could be ground was accomplished by placing the bulk material inside a heavy plastic glove box where it was broken into pebbles. The pebble-sized material was then crushed by an iron mortar and pestle to particles ranging from dust to small sand-sized granules. A charge of approximately 125 g was loaded into a cryogenic grinding device. Since no difference was noted between cryogenic and room temperature grinding, the operation was performed at room temperature to avoid the introduction of large amounts of water into the sample.

The loading and unloading of the grinder was conducted in plexiglass boxes both to minimize contamination and to contain radioactive dust particles. After each grinding, the product was sieved by a 120 and a 200 mesh brass sieve set. All materials passing the sieve were set aside and the remaining coarse material reprocessed. This procedure was repeated until only a few grams of coarse material remained for discarding. The sieved material was blended in a twin V cone blender according to a prescribed procedure and then stored in a clean polyethylene container for final packaging.

2. Uranium Assay Reference Material

SRM 950b, a re-issue and replacement reference material for the exhausted SRM 950a, is now available to licensees, DOE contractors, and other qualified users. The new SRM is certified to be 99.968 ± 0.020 percent uranium oxide (U_3O_8) and is intended as a reference material of known uranium content. The certified data is based on material ignited at 800 °C for one hour in an open crucible in a muffle furnace and cooled in a desiccator. It is critical that the materials be freshly ignited in the prescribed manner to obtain accurate assay results.

SRM 950b was certified through a cooperative research effort by NBS and NBL. The original assay measurements at NBS were performed coulometrically and the assay at NBL by the NBL titrimetric method. Since the results differed by more than 0.03 percent, NBS thoroughly investigated the coulometric and

titrimetric methods. After several modifications of the coulometric method, SRM 950b and SRM 960a, uranium metal, were analyzed. The assay results in Table 2 for SRM 960 agreed very well with the certified value of 99.975 ± 0.017 percent and is indicative of a high degree of control on a sample of known uranium content. The data (Table 2) for NBS and NBL measurements of SRM 950b agreed to within 0.01 percent and satisfied all statistical tests of a homogeneous group which would permit certification at the desired level of accuracy. The uncertainty of ± 0.02 percent in the uranium assay of SRM 950b includes an allowance for small inter-laboratory differences in the NBL method and the slightly larger imprecision of the coulometric method.

The isotopic composition of SRM 950b necessary to calculate the atomic weight was determined by the joint efforts of NBS, the Goodyear Atomic Corporation and the Paducah Gaseous Diffusion Plant, Isotope Measurements Laboratory. The NBS measurements are based upon a point calibration using calibration mixes of known isotopic composition. These calibration mixes were prepared by gravimetrically blending aliquots of highly enriched and depleted uranium. The isotopic abundances of the SRM were determined by thermal ionization mass spectrometry and are shown in Table 3. Goodyear Atomic Corporation and Paducah Gaseous Diffusion Plant used uranium hexafluoride mass spectrometry to determine the ^{235}U composition. Calibration was achieved using two completely different sets of standards. The Goodyear and Paducah values are 0.71970 and 0.71972 atom percent, respectively. Combining the data of the three laboratories yields a set of values from which an atomic weight of 238.0289 is calculated.

During the next quarter it is anticipated that progress will be made in the following areas:

1. Certification of a ^{238}U spike.

2. Resolution of the apparent systematic difference in NBS-CBNM ^{238}Pu measurements.

3. Assay by both coulometry and the NBL titrimetric method of SALE enriched and depleted uranium metal.

4. Isotopic abundance determination of SALE enriched and depleted uranium metal.

Investigators for this task are: E. Garner, L. Machlan, J. Moody and J. Maples.

Table 1. ^{238}Pu Atomic Composition for AS-76
(Corrected to 11-1-77)

Sample	CBNM (GEEL)	NBS	% Diff.
E131	0.204	0.205	0.5
D212	0.798	0.792	0.8
S11	0.912	0.904	0.9
M31	1.651	1.652	0.06

Table 2. Uranium Assay Results

SRM	Assays	Found (&)	σ (%)
960	14	99.971	0.005
950a	4[a]	99.938	0.003
950b	8[a]	99.969	0.006
950b[b]	16	99.967	0.016
950b[c]	6	99.958	0.022

[a] Ignition in muffle furnace

[b] Determined by NBL

[c] Determined by Coulometry

Table 3. Isotopic Composition of SRM 950b

	^{234}U	^{235}U	^{238}U
NBS	0.0053	0.7200	99.2747
	± 0.0001[a]	± 0.0006[a]	± 0.0007[a]
Best Value	0.0053	0.7198	99.2749
	± 0.0001[a]	± 0.0004[a]	+ 0.0005[a]

Atomic Weight = 238.0289

[a]Estimated Uncertainty

Task III: STANDARDIZATION OF NON-DESTRUCTIVE ASSAY METHODOLOGY FOR NUCLEAR MATERIALS

A. Gamma and X-Ray Spectrometry

The activities reported on last quarter have continued with minor difficulties.

Final drawings and concepts for the Segmented Gamma Scan Unit have been agreed upon and J. Langland of the Technical Support Group for the Center for Analytical Chemistry is to act as a liaison with the NBS shops. This unit will be interfaced with the multi-task, multi-user, computer-based multichannel analyzing system and be capable of supporting either one or two horizontal mounted Ge(Li) detectors for vertical scanning. In addition to the vertical scanning capabilities, the unit will also be able to accommodate a vertical mounted Ge(Li) detector for horizontal scanning and K-edge absorption measurements.

In order to compile an archival gamma-ray spectrum library of known ^{235}U enrichment levels, counting standards were prepared from the NBS SRM Uranium Isotopic Standards (see Table 4). These standards were prepared with the help of L. A. Machlan and J. S. Maples of the Inorganic Analytical Research Division. The U_3O_2 stock material was fired at 800 °C for 1 hour, then a nominal 300 mg of material was transferred into thin window Lucite holders and sealed. Examples of the data obtained from these standards are given in Tables 5-7. In addition to the counting standards, a SNM gamma-ray nuclide library has been compiled for NDA.

Although the work on the joint NBS-Euratom U_3O_8 NDA standards has been delayed slightly due to difficulties with container fabrication, the SNM import license has been obtained from NRC.

Finally, the INMM Task Group 9.3 of the ANSI Standards Committee N15, with participation by NBS, has completed writing its standard, "Physical Standards for Nondestructive Assay (NDA)," and is now having it circulated for review.

The investigator for the gamma and x-ray spectrometry portion of the task is S. Carpenter.

Table 4

Uranium Isotopic Counting Standards

SRM#	U_3O_8 Wt(mg)	^{235}U Atom Percent	^{238}U Atom Percent
U-0002	300.93	0.01755	99.9823
U-005	340.71	0.4895	99.504
U-010	311.44	1.0037	98.984
U-015	333.48	1.5323	98.443
U-020	313.54	2.038	97.933
U-030	315.75	3.046	96.915
U-050	325.69	5.010	94.915
U-100	325.93	10.190	89.704
U-500	319.29	4 .696	49.711
U-930	330.93	93.336	5.380
U-970	332.76	97.663	0.5229
Depleted Primary	317.63	0.0004	99.9996
Enriched Primary	316.06	99.8195	0.0789
950a	311.72	0.7198	99.2749
Belgium Congo Pitchblende	326.30	0.720	99.274

Table 5

SRM U-0002 Counting Standard

```
**********************************************************************
*****************  11 MAY 1978  5:20:57 PM   *****************
**********************************************************************

GAMMA-X TEST

SAMPLE DATE:    7APR78   14:54
SAMPLE IDENTIFICATION:  U-0002
TYPE OF SAMPLE:  U3O8
SAMPLE QUANTITY:    300.9330          UNITS:  MG
SAMPLE GEOMETRY:  0.5 CM
EFFICIENCY FILE NAME:  EFF.TAB4
**********************************************************************
                                    *                                 -
ACQUISITION DATE: 10APR78  757:24   * FWHM(1332)            2.041
PRESET TIME(LIVE):       3600. SEC  * SENSITIVITY:          5.000
ELAPSED REAL TIME:       3617. SEC  * SHAPE PARAMETER      20.0 %
ELAPSED LIVE TIME:       3600. SEC  * NBR ITERATIONS:       10.
                                    *
**********************************************************************
                                    *
DETECTOR: ORTEC GAMMA-X             * LIBRARY NUCL.LIB
DATE CALIBRATED:    7APR78 1423:34  * ENERGY TOLERANCE:   1.250KV
KEV/CHNL:        0.2799662          * HALF LIFE RATIO:     8.00
OFFSET:          0.2566251 KEV      * ABUNDANCE LIMIT    80.00%
Q. COEFF.       -5.324E-08 KEV/C**2 *
                                    *
**********************************************************************

                ENERGY WINDOW      0.817 TO   1146.105

PK E IT CHANNEL LEFT PW     BKGND    FWHM      AREA  ENERGY   CTS/SEC    %ERR

 1 0  0   24.96   19 15      2141.   1.05      2029.    7.24 5.635E-01   3.9
 2 0  0   46.51   43  9      3166.   1.08       815.   13.28 2.264E-01  10.4
FIT=   3.462922E 00
 3 0 10   57.39   51 45      4160.   1.50      4499.   16.32 1.250E 00   2.5
 4 0 10   70.13   51 45      6184.   2.17      2143.   19.89 5.953E-01   5.6
 5 0  0  175.39  171 10      7694.   0.96       365.   49.36 1.013E-01  14.4
 6 0  0  189.05  185  9      7386.   1.02       347.   53.18 9.642E-02  35.4
 7 0  0  224.93  219 13     10946.   0.90     47748.   63.23 1.326E 01   0.6
 8 0  0  296.14  291 11      9944.   0.78       931.   83.16 2.586E-01  15.5
 9 0  0  330.44  318 25     19416.   1.10     96516.   92.76 2.681E 01   0.4
10 0  0  350.56  344 14     10707.   0.99     17333.   98.40 4.815E 00   1.1
FIT=   2.955978E 00
11 0 10  395.88  388 27      5271.   1.53      7141.  111.08 1.984E 00   1.9
12 0 10  401.99  388 27      2938.   0.86      4149.  112.79 1.153E 00   2.4
13 0 10  408.14  388 27      4521.   1.53      2701.  114.51 7.504E-01   4.0
14 0  0  467.63  464  9      2900.   0.95       378.  131.17 1.049E-01  20.8
15 0  0  662.39  655 14      2802.   1.01       944.  185.68 2.623E-01   8.6
16 0  0  921.50  916 14      1822.   0.96       538.  258.20 1.494E-01  12.0
17 0  0 2653.47 2646 15       594.   1.70       162.  742.76 4.496E-02  22.7
18 0  0 2737.88 2730 16       648.   1.60       630.  766.37 1.751E-01   7.0
19 0  0 3576.07 3565 21       349.   1.69      1491. 1000.75 4.143E-01   3.1

PEAK SEARCH COMPLETED
```

Table 6

SRM U-500 Counting Standard

```
**********************************************************************
*****************  11 MAY 1978  5:24:29 PM   *****************
**********************************************************************

GAMMA-X TEST

SAMPLE DATE:    7APR78   14:54
SAMPLE IDENTIFICATION:  U-500
TYPE OF SAMPLE:  U3O8
SAMPLE QUANTITY:    319.2900          UNITS:  MG
SAMPLE GEOMETRY:  0.5 CM
EFFICIENCY FILE NAME:  EFF.TABM
**********************************************************************
                                    *
ACQUISITION DATE: 11APR78 1217:14   * FWHM(1332)            2.042
PRESET TIME(LIVE):       3600. SEC  * SENSITIVITY:          5.000
ELAPSED REAL TIME:       3798. SEC  * SHAPE PARAMETER :  20.0 %
ELAPSED LIVE TIME:       3600. SEC  * NBR ITERATIONS:     10.
                                    *
**********************************************************************
                                    *
DETECTOR: ORTEC GAMMA-X             * LIBRARY:NUCL.LIBM
DATE CALIBRATED:    7APR78 1423:34  * ENERGY TOLERANCE:   1.250KV
KEV/CHNL:         0.2800000         * HALF LIFE RATIO:    8.00
OFFSET:           0.2566000 KEV     * ABUNDANCE LIMIT:   80.00%
Q. COEFF. :    -5.324E-08 KEV/C**2  *
                                    *
**********************************************************************

                ENERGY WINDOW      0.817 TO   1146.243

PK E IT CHANNEL LEFT PW    BKGND    FWHM     AREA  ENERGY  CTS/SEC    %ERR

 1 0  0   23.55   19 11    26012.   0.57    32267.    6.85 8.963E 00  0.9
 2 0  0   31.75   29 11    59909.   0.80     1793.    9.15 4.980E-01 19.5
FIT=   1.230843E 02
 3 0  7   45.33   40 35    36285.   1.13    20558.   12.95 5.710E 00  1.5
 4 0  7   56.44   40 35    36262.   1.06   182401.   16.06 5.067E 01  0.3
 5 0  7   66.97   40 35    41418.   1.21    61180.   19.01 1.699E 01  0.6
 6 0  0   90.10   82 15    72583.   0.89    44738.   25.48 1.243E 01  1.0
 7 0  0  151.54  147 10    36035.   1.69     3122.   42.69 8.672E-01  8.8
 8 0  0  188.99  183 13    51223.   0.88    99224.   53.17 2.756E 01  0.5
 9 0  0  208.18  203 12    46271.   0.92    17655.   58.54 4.904E 00  1.9
10 0  0  225.09  219 14    63623.   0.93    27640.   63.28 7.678E 00  1.4
FIT=   9.704030E 00
11 0 10  258.69  250 34    58739.   1.34    21254.   72.69 5.904E 00  1.8
12 0 10  265.36  250 34    58491.   1.41    12310.   74.55 3.420E 00  2.9
13 0 10  273.86  250 34   116381.   2.69    14145.   76.93 3.929E 00  3.5
14 0  0  298.15  284 23   133748.   0.94   413210.   83.73 1.148E 02  0.2
FIT=   9.028301E 02
15 0  6  320.23  313 45    29622.   0.97   193759.   89.92 5.382E 01  0.3
16 0  6  332.02  313 45    32231.   1.12   284642.   93.22 7.907E 01  0.2
17 0  6  337.06  313 45    31016.   1.03   330499.   94.63 9.181E 01  0.2
18 0  6  350.53  313 45    24416.   0.99   474540.   98.40 1.318E 02  0.2
19 0  0  364.46  361  9    32078.   0.91    22199.  102.30 6.166E 00  1.3
```

```
FIT=   1.221407E 02
20 0  4  375.46  369 51    33500.  1.46    88426.  105.38 2.456E 01  0.4
21 0  4  388.67  369 51    29060.  1.43    14628.  109.08 3.990E 01  0.3
22 0  4  395.89  369 51    31562.  1.48   205094.  111.10 5.697E 01  0.3
23 0  4  408.28  369 51    30399.  1.51    78752.  114.57 2.188E 01  0.5
24 0  0  430.83  425 13    41634.  0.97    42499.  120.88 1.181E 01  0.8
25 0  0  445.41  441 10    29424.  0.98     2614.  124.96 7.260E-01  9.5
FIT=   1.000101E 00
26 0  5  477.78  468 21    15433.  0.85      992.  134.02 2.757E-01 18.0
27 0  5  482.76  468 21    15032.  0.90     2608.  135.70 7.244E-01  6.9
28 0  0  512.30  497 23    51302.  1.03   457938.  143.69 1.272E 02  0.2
29 0  0  538.02  533 11    19943.  1.13     2580.  150.89 7.167E-01  8.0
30 0  0  582.50  575 15    24641.  1.06   204526.  163.34 5.681E 01  0.2
31 0  0  628.04  623 11    16999.  0.65      670.  176.09 1.862E-01 27.8
32 0  0  662.33  647 23    29383.  1.07  2140087.  185.69 5.945E 02  0.1
33 0  0  695.37  679 24    23811.  0.99    18687.  194.94 5.191E 00  1.4
34 0  0  730.30  705 35    13602.  1.09   209644.  204.71 5.823E 01  0.2
FIT=   1.457461E 00
35 0  7  767.98  763 20     2748.  1.38     1095.  215.26 3.043E-01  7.4
36 0  7  777.39  763 20     2373.  1.22     1424.  217.89 3.955E-01  5.5
37 0  0  789.93  783 14     4181.  1.14     3858.  221.40 1.072E 00  2.9
38 0  0  833.39  828 12     3092.  1.05     1867.  233.57 5.185E-01  4.8
FIT=   2.587051E 00
39 0  9  845.75  840 36     2451.  1.42     1670.  237.03 4.640E-01  4.9
40 0  9  851.52  840 36     1833.  1.08     2107.  238.64 5.852E-01  3.6
41 0  9  859.55  840 36     1736.  1.04     2007.  240.89 5.574E-01  3.7
FIT=   3.163573E-01
42 0  9  880.88  875 21     1976.  1.20     1169.  246.86 3.248E-01  6.1
43 0  9  889.02  875 21     2412.  1.52      585.  249.14 1.626E-01 12.6
FIT=   4.272122E 00
44 0  3  983.69  975 33     1872.  1.23     4311.  275.64 1.197E 00  2.1
45 0  3  995.92  975 33     1827.  1.23     6592.  279.06 1.831E 00  1.5
FIT=   5.276130E 00
46 0  6 1013.71 1008 51     2081.  1.67      826.  284.04 2.295E-01  8.5
47 0  6 1039.27 1008 51     2054.  1.58     2379.  291.20 6.609E-01  3.4
48 0  6 1051.40 1008 51     2136.  1.50      570.  294.59 1.583E-01 12.2
49 0  0 1234.60 1228 13     1056.  1.31     1428.  345.86 3.966E-01  4.2
FIT=   1.819999E 00
50 0 10 1314.42 1304 28      531.  1.35     1222.  368.20 3.395E-01  3.9
51 0 10 1324.11 1304 28      640.  2.13      903.  370.91 2.507E-01  5.2
52 0  0 1384.43 1377 15      722.  1.31     3056.  387.79 8.489E-01  2.2
53 0  0 2082.83 2075 18      423.  1.50      480.  583.22 1.332E-01  7.6
54 0  0 2737.22 2729 18      293.  0.99      369.  766.28 1.025E-01  8.4
55 0  0 3575.90 3566 19      183.  1.62      728. 1000.83 2.022E-01  4.5


 PEAK SEARCH COMPLETED
```

Table 7

SRM U-970 Counting Standard

```
**********************************************************************
*****************  11 MAY 1978  5:30:26 PM   *****************
**********************************************************************

GAMMA-X TEST

SAMPLE DATE:    7APR78   14:54
SAMPLE IDENTIFICATION:  U-970
TYPE OF SAMPLE:  U3O8
SAMPLE QUANTITY:    332.7600          UNITS:  MG
SAMPLE GEOMETRY:  0.5 CM
EFFICIENCY FILE NAME:  EFF.TABN
**********************************************************************
                                      *
ACQUISITION DATE: 12APR78  744:20   * FWHM(1332)          2.042
PRESET TIME(LIVE):      3600. SEC   * SENSITIVITY:        5.000
ELAPSED REAL TIME:      4023. SEC   * SHAPE PARAMETER :  20.0 %
ELAPSED LIVE TIME:      3600. SEC   * NBR ITERATIONS:    10
                                      *
**********************************************************************
                                      *
DETECTOR: ORTEC GAMMA-X               * LIBRARY:NUCL.SFLIB
DATE CALIBRATED:    7APR78 1423:34  * ENERGY TOLERANCE:  1.250KV
KEV/CHNL:        0.2800000          * HALF LIFE RATIO:   8.00
OFFSET:          0.2566000 KEV      * ABUNDANCE LIMIT:  80.00%
Q. COEFF. :    -5.324E-08 KEV/C**2  *
                                      *
**********************************************************************

                ENERGY WINDOW      0.817 TO   1146.243

PK E IT CHANNEL LEFT PW     BKGND    FWHM      AREA  ENERGY  CTS/SEC    %ERR

FIT=   1.484438E 02
 1 0 10   23.02   20 18      9933.   1.04     59855.    6.70 1.663E 01  0.5
 2 0 10   29.78   20 18     63853.   2.54     91291.    8.60 2.536E 01  0.5
FIT=   2.566566E 02
 3 0  5   45.14   39 37     77840.   1.08     56458.   12.90 1.568E 01  0.8
 4 0  5   56.25   39 37     77159.   1.06    558215.   16.01 1.551E 02  0.2
 5 0  5   66.74   39 37     76540.   1.14    172396.   18.94 4.789E 01  0.3
 6 0  0   89.99   82 15    148980.   0.89     94172.   25.45 2.616E 01  0.7
 7 0  0  186.85  144 52    377033.   0.90    380273.   52.57 1.056E 02  0.3
 8 0  0  208.10  203 12     90122.   0.92     36738.   58.52 1.020E 01  1.3
 9 0  0  227.01  223  9     80097.   0.54      9411.   63.82 2.614E 00  4.4
FIT=   1.903587E 01
10 0 10  258.65  251 33    105727.   1.26     42004.   72.67 1.167E 01  1.2
11 0 10  265.37  251 33    130346.   1.49     29456.   74.56 8.182E 00  1.8
12 0 10  274.07  251 33    192218.   2.29     30179.   76.99 8.381E 00  2.1
13 0  0  298.08  284 23    264011.   0.96    884189.   83.72 2.456E 02  0.1
FIT=   1.656727E 03
14 0  3  320.14  313 45     60715.   0.99    430714.   89.89 1.19FE 02  0.2
15 0  3  332.19  313 45     65949.   1.03    540447.   93.26 1.501E 02  0.2
16 0  3  337.00  313 45     63995.   1.03    703320.   94.61 1.954E 02  0.1
17 0  3  350.45  313 45     58914.   1.00   1029672.   98.38 2.860E 02  0.1
18 0  0  364.39  361  9     68392.   0.91     46569.  102.28 1.294E 01  0.9
FIT=   8.773287E 00
```

```
FIT=   1.977287E 02
19 0  4  375.37  369 48     71662.   1.47    193114.   105.35 5.364E 01   0.3
20 0  4  388.61  369 48     63116.   1.43    307665.   109.06 8.546E 01   0.2
21 0  4  395.81  369 48     69203    1.48    471309    111.09 1.198E 02   0.2
22 0  4  408.21  369 48     67736.   1.49    167363.   114.55 4.649E 01   0.3
23 0  0  430.71  424 14     96163.   0.99    143820.   120.85 3.995E 01   0.4
24 0  0  444.98  441 10     62098.   1.05      4999.   124.84 1.389E 00   7.2
FIT=   2.201102E 00
25 0  8  477.31  469 19     26569.   0.78      1867.   133.89 5.185E-01  12.6
26 0  8  483.43  469 19     31829.   0.90      4634    135.61 1.287E 00   5.6
27 0  0  512.24  497 23    108221.   1.04    981207.   143.67 2.726E 02   0.1
28 0  0  538.13  533 11     42052.   1.05      6026.   150.92 1.674E 00   5.0
29 0  0  582.43  575 15     53593.   1.06    440745.   163.32 1.224E 02   0.2
FIT=   1.334501E 00
30 0  3  621.25  612 20     25289.   1.29      3645.   174.19 1.012E 00   6.4
31 0  3  626.87  612 20     25453.   1.25      3102.   175.76 8.617E-01   7.5
FIT=   1.087724E 06
32 0 10  640.57  634 43     20993.   1.14      2452.   179.59 6.812E-01   8.6
33 0 10  650.98  634 43     21613.   1.27     23500.   182.51 6.528E 00   1.1
34 0 10  652.37  634 43     29250.   1.74   1372983.   182.90 3.814E 02   0.1
35 0 10  662.35  634 43     16631.   1.08   3291346.   185.69 9.143E 02   0.1
36 0  0  694.84  679 24     41250.   1.06     49604.   194.79 1.378E 01   0.7
37 0  0  730.33  705 35     48304.   1.09    450692.   204.72 1.252E 02   0.2
FIT=   1.466928E 00
38 0  5  767.95  761 22      5968.   0.96      1455.   215.25 4.041E-01   8.0
39 0  5  777.45  761 22      6878.   1.01      2149.   217.91 5.969E-01   5.9
40 0  0  789.88  783 15     13466.   1.11      8056.   221.39 2.238E 00   2.3
FIT=   2.021186E 00
41 0  7  833.34  824 43      5943.   1.10      3595.   233.55 9.985E-01   3.5
42 0  7  845.71  824 43      6602.   1.29      3816.   237.02 1.060E 00   3.4
43 0  7  851.40  824 43      5704.   1.08      8278.   238.61 2.299E 00   1.7
44 0  7  859.66  824 43      5598.   1.05      4519.   240.92 1.255E 00   2.9
45 0  0  883.44  875 20     14390.   1.13      4091.   247.58 1.136E 00   4.4
46 0  0  902.17  897 12      8780.   1.39       747.   252.82 2.075E-01  18.1
47 0  0  916.69  911 11      7873.   0.99       929.   256.88 2.580E-01  13.9
FIT=   1.190113E 01
48 0  3  983.68  976 31      5695.   1.21      9857.   275.64 2.738E 00   1.5
49 0  3  995.92  976 31      5425.   1.20     15954.   279.06 4.432E 00   1.0
50 0  0 1013.11 1007 13      6832.   1.27      2434.   283.87 6.761E-01   5.2
FIT=   3.890292E 00
51 0  5 1039.25 1030 34      5015.   1.64      6334.   291.19 1.759E 00   2.0
52 0  5 1051.55 1030 34      5014.   1.75      2407.   294.63 6.686E-01   4.6
53 0  5 1058.74 1030 34      5014.   1.65      1370.   296.64 3.806E-01   7.8
54 0  0 1071.49 1064 15      5213.   1.27      2060.   300.21 5.721E-01   5.4
55 0  0 1164.99 1161 10      2703.   0.88       366.   326.38 1.016E-01  20.8
56 0  0 1174.74 1170 14      3642.   0.84       447.   329.11 1.243E-01  19.7
57 0  0 1201.31 1196 12      1976.   1.14       523.   336.55 1.454E-01  12.8
58 0  0 1236.13 1225 25      4106.   1.25      3977.   346.29 1.105E 00   2.8
FIT=   4.585358E 00
59 0  8 1314.82 1305 27      1670.   1.52      2984.   368.31 8.288E-01   2.7
60 0  8 1324.28 1305 27      1379.   1.80      2773.   370.96 7.702E-01   2.7
61 0  0 1384.54 1377 15      1181.   1.21      6967.   387.83 1.935E 00   1.4
62 0  0 1431.41 1419 19       589.   0.86        90.   400.94 2.512E-02  39.4
63 0  0 1544.52 1538 13       232.   0.78       148.   432.59 4.106E-02  16.7
64 0  0 1614.45 1607 18       277.   1.33        57.   452.16 1.585E-02  43.3
65 0  0 1824.13 1818 16       204.   1.33       475.   510.84 1.319E-01   6.3
66 0  0 2082.84 2071 21        13.   1.50      1703.   583.22 4.732E-01   2.4
67 0  0 2597.72 2590 17        17.   1.53       349.   727.26 9.694E-02   5.6
68 0  0 3074.22 3061 28        32.   1.51       178.   860.53 4.938E-02   8.7


 PEAK SEARCH COMPLETED
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B. Calorimetry

An operating heat-flow calorimeter has been received on a loan basis from the Mound Laboratory. This calorimeter is designed for power measurements in the range 0-5W. Experience gained through its operation will provide information necessary to guide the choice of calorimeter design for the NBS installation. We are now in the process of installing the calorimeter in an existing constant-temperature bath and instrumenting it. A group of standard resistors supplied with the calorimeter for precise current and potential measurements has been submitted for NBS calibration.

Four encapsulated plutonium standard heat sources were also obtained on loan from Mound. These have nominal powers of 4.0, 1.0, 0.25, and 0.25 W and will be compared in the initial measurements in the Mound heat-flow and NBS Bunsen ice calorimeters.

Investigators for the Calorimetry portion of the task are D. Ditmars and J. Colwell.

C. Resonance Neutron Radiography-Linac Produced Neutrons

The principle of resonance neutron radiography as a possible reference method for nondestructive assay was demonstrated quantitatively for the first time. A basic system has been established which will be refined and embellished as equipment on order or under construction is incorporated. However, measurements with the present system showed very satisfactory neutron background, neutron intensity, and energy resolution. A measurement, therefore, was undertaken on a tungsten sample which exhibits low energy resonance structure typical of the fissile materials. The tungsten was placed inside an aluminum can along with a tape cassette to confuse the radiograph and the can was stuffed full of tissue paper to keep the sample stationary. A narrow resonance in tungsten at 7.5 eV was selected for performing the radiography. Transmission measurements as a function of position were taken over about a 10-hour period using the NBS linac neutron source. The neutron energy was measured by time-of-flight and the neutron position by pulse height.

A map of transmission at the tungsten resonance clearly showed the shape of the ".014" thick tungsten plate. The integration of the transmission over the shape yielded a tungsten mass which agreed with the tungsten mass determined by its weight within the accuracy of the nuclear data on the tungsten resonance. The expected accuracy of the present system for the tungsten plate was about ±5% aside from the nuclear data uncertainties. Thus, we presently have a rudimentary

facility with the capability to radiograph unknown objects, determine by means of their low energy neutron resonance structure the existence of an isotope or element, define the shape of the object, and compute the mass of the isotope in the object.

The study of chemical and physical effects continues with the expected release of two publications investigating the influence of molecular vibrations on the nuclear measurements which might have application in the assay of nuclear materials.

During the next quarter, better mechanical components of the radiography facility will be added. The computer is expected to arrive and interfacing to the experiment will begin. Also, software development necessary for computer data acquisition and analysis, will begin.

Investigators for the resonance neutron radiography with linac-produced neutrons portion of the task are C. Bowman, R. Schrack and J. Behrens.

D. Resonance Neutron Tomography-Reactor Produced Neutrons

The design of the reactor facility has been completed. A neutron access port, through which the resonance neutron tomography reentrant tube will be inserted into the NBS Research Reactor core, has been assigned to the Resonance Neutron Tomography Project. The reentrant tube has been engineered and designed and the system has been approved by the Hazards Evaluation Committee and the safety analysis completed. With little or no modification, this tube design can be employed to meet the Resonance Neutron Tomography System requirements. Engineering drawings have been obtained and are under review prior to this fabrication in the NBS Reactor Shop.

The design of the tomography scanning hardware is moving ahead, off-the-shelf rotational and traversal hardware has been located. These systems are manufactured by Velmex Incorporated and are stepping motor driven, thus making them compatible with computer control. Tentative system specifications have been established.

Tomographic Image Reconstruction Hardware is also being assembled. To achieve the desired real-time tomographic results, it is necessary to have data acquisition hardware capable of three-dimensional image reconstruction containing a large number of pixels (picture elements). To optimize cost effectiveness, it was decided to acquire such a system on a cost sharing basis with three other groups within the Reactor Radiation Division, i.e. the Neutron Radiography, and two Nuetron Scattering groups. An advanced state-of-the-art data acquisition system based on a DEC VAX 11/780 processor has been requested. This system

contains a 128 K memory and utilizes 32 bit words. System purchase is pending Department of Commerce approval.

System Performance Simulation plans have now been firmed up. An industrial x-ray tomography system has been designed and fabricated by personnel of the Nondestructive Testing Group of the Los Alamos Scientific Laboratory. In addition, simulation software has been developed in which performance can be predicted by varying system parameters, e.g. geometry, source collimation, waste container size, etc.

A solicited proposal has been received from the Los Alamos Scientific Laboratory to run a simulation routine for neutrons of the proposed NBS Resonance Neutron Tomography Facility for the characterization of the isotopic geometry of phantom nuclear waste containers. Variable parameters to be embodied in this study include:

(1) Isotopic content

(2) Isotopic geometry

(3) Container size

(4) Packing material

(5) Neutron beam collimation

It is envisioned that this simulation experiment will provide insight into the NBS system capabilities, limitation and design.

We propose to award this contract to LASL. This is cost-effective in that the simulation software exists and is working at LASL which will drastically reduce the experimental work necessary at the NBS to get on with the job at hand.

The investigator for the resonance neutron tomography using reactor produced neutrons portion of the task is D. Garrett.

Task IV: FEASIBILITY STUDY ON THE USE OF ACCURATE INFRARED THERMOGRAPHY TECHNIQUES FOR NUCLEAR MATERIALS

Phase 10 of the feasibility study, gathering and evaluating of information about commercially available thermographic instrumentation is now finished. A brief statement of our findings was given at the NRC Mid-Year Review on March 30. We find that most of the instrumentation approaches near the state-of-the-art performance. It is unlikely that any significant improvement in measurement sensitivity or accuracy will occur in the near future unless there is a break-thru in the state-of-the-art.

During the third quarter, the emphasis has been on Phase II,

a detailed facility analysis. We have concentrated on two cases; the Rocky Flats Plant as an example of a typical existing facility, and the proposed Westinghouse-Anderson Recycle Fuel Plant as an example of a typical facility of the future. Our analysis so far indicates that there are probably very few locations in either facility where thermography could be utilized advantageously for quantitative holdup measurements. We will extend our analysis to other facilities during the fourth quarter.

Investigators for this task are Harvey Marshak and Martin Reilly.

Task V: STANDARDIZATION OF BULK MEASUREMENT METHODOLOGY IN NUCLEAR FUEL CYCLE PLANTS

A. Field Calibration of In-Plant Accountability Tanks

In the effort concerned with volume calibration of input accountability tanks, we have secured the cooperation of the DOE-Savannah River facility to carry out actual field tests in an operating reprocessing facility. The data for the tank at the Savannah River Plant has been further refined. A new algorithm relating water calibration to the calibration of a tank containing process fluid at the appropriate temperature is being developed.

Savannah River Plant personnel have calibrated an evaporation tank using procedures used by NBS personnel on the input accountability tank calibration and with consultation by NBS personnel. The data will be analyzed with the cooperation of NBS.

A seminar on volume calibration was conducted at the Savannah River Plant which was attended by DOE and DuPont personnel.

A paper, "the Application of an Improved Calibration System to the Calibration of Accountability Tanks," is being prepared for presentation at the IAEA International Symposium on Nuclear Materials Safeguards, Vienna, Austria, October 2-6, 1978 and for inclusion in the Symposium proceedings. The paper will outline the procedures used in volume calibration using volumetric transfer standards and differential electromanometers. The data for the calibration of the Savannah River Plant input accountability tank will be used to illustrate the precision of the calibration system in the field.

Figure 1 is a drawing of a tank similar to the SRP input accountability tank. In the treatment of the calibration data, the tank was divided into four regions: I) the region of the cooling coils; II) the transition region at the top of the coils; III) the region above the transition region but below the headers (sealed cylindrical sections of tubing perpendicular to the cooling water lines, occupying a volume

of approximately 2 liters, not shown in the figure) and IV) the region above the headers. The linear calibration equations developed for the four regions given are shown below:

Region I (coils)

$$V(m^3) = 0.12345 + 0.000426127\ P(Pa)$$

Region II (transition)

$$V(M^3) = -0.11394 + 0.000447373\ P(Pa)$$

Region III (above transition, below headers)

$$V(m^3) = -0.37183 + 0.000465534\ P(Pa)$$

Region IV (above transition, above headers)

$$V(m^3) = -0.37067 + 0.000465356\ P(Pa)$$

These equations relate volume, V, in cubic meters, to pressure, P, in pascals. Both V and P are "reduced" to a temperature of 25 °C. The coefficients are subject to further refinement.

The fit of the calibration data to the calibration equations is illustrated by Table 8 where:

V_{cN} is the volume calculated using the calibration equations;

$\%_N$ is equal to 100 x $(V_{cN} - V)/V$, the "relative residual";

V_{cSR} is the volume calculated using calibration equations developed by SRP for their 1971 calibration; and $\%_{SR}$ is the relative residual using the SRP equations.

In regions I, III and IV, the maximum absolute value of the relative residuals is 0.049%. In region II, the transition region, a linear equation is not an adequate representation of the calibration data. SRP uses region II of the tank for accountability purposes. Comparison of the values in the $\%_N$ column with those in the $\%_{SR}$ column gives an indication of the improvement in calibration precision gained by the use of the improved calibration system.

Table 8

		V(liters)	V_{cN}-V	$\%_N$	V_{cSR}-V	$\%_{SR}$
	1	378.84				
	2	757.67				
	3	1136.46				
	4	1515.23	-0.20	-0.013	+8.54	+0.56
	5	1894.01	-0.24	-0.013	+4.29	+0.23
	6	2272.72	+1.12	+0.049	+1.42	+0.06
	7	2651.54	+0.31	+0.012	-3.58	-0.14
Region I	8	3030.29	-0.72	-0.024	-8.81	-0.29
	9	3409.19	+0.44	+0.013	-11.87	-0.35
	10	3788.05	+0.07	+0.002	-16.45	-0.43
	11	4166.93	-0.79	+0.019	-21.51	-0.52
	12	4545.79	-8.04		-29.52	
	13	4924.70	+2.33		-29.20	
	14	5303.59	+13.34		-28 26	
Region II	15	5682.48	+12.48		-38.88	
	16	6061.40	-2.07		-52.84	
	17	6440.25	-18.05		-88.19	
	18	6819.13	+0.50	+0.007	-54.79	-0.80
	19	7197.98	+0.07	+0.001	-59.93	-0.83
	20	7576.84	0.00	0.000	-64.72	-0.85
	21	7955.65	-0.21	-0.003	-69.65	-0.88
Region III	22	8334.53	-0.62	-0.007	-74.78	-0.90
	23	8713.40	-0.43	-0.005	-79.30	-0.91
	24	9092.41	+0.15	+0.002	-83.46	-0.92
	25	9471.35	+0.46	+0.005	-87.87	-0.93
	26	9850.20	+0.07	+0.0007	-92.97	-0.94
	27	10229.08				
	28	10607.93				
	29	10986.76	+0.17	+0.0016	-104.71	-0.95
	30	11365.62	+0.53	+0.0047	-109.03	-0.96
Region IV	31	11744.46	-0.40	-0.003	-114.63	-0.98
	32	12123.43	-0.28	-0.0023	-119.21	-0.98
	33	12502.38	-0.01	-0.00008	-123.63	-0.99
	34	12881.28				
	35	13260.22				
	36	13639.14				

Investigators for this portion of the task are Frank Jones and John Hauser.

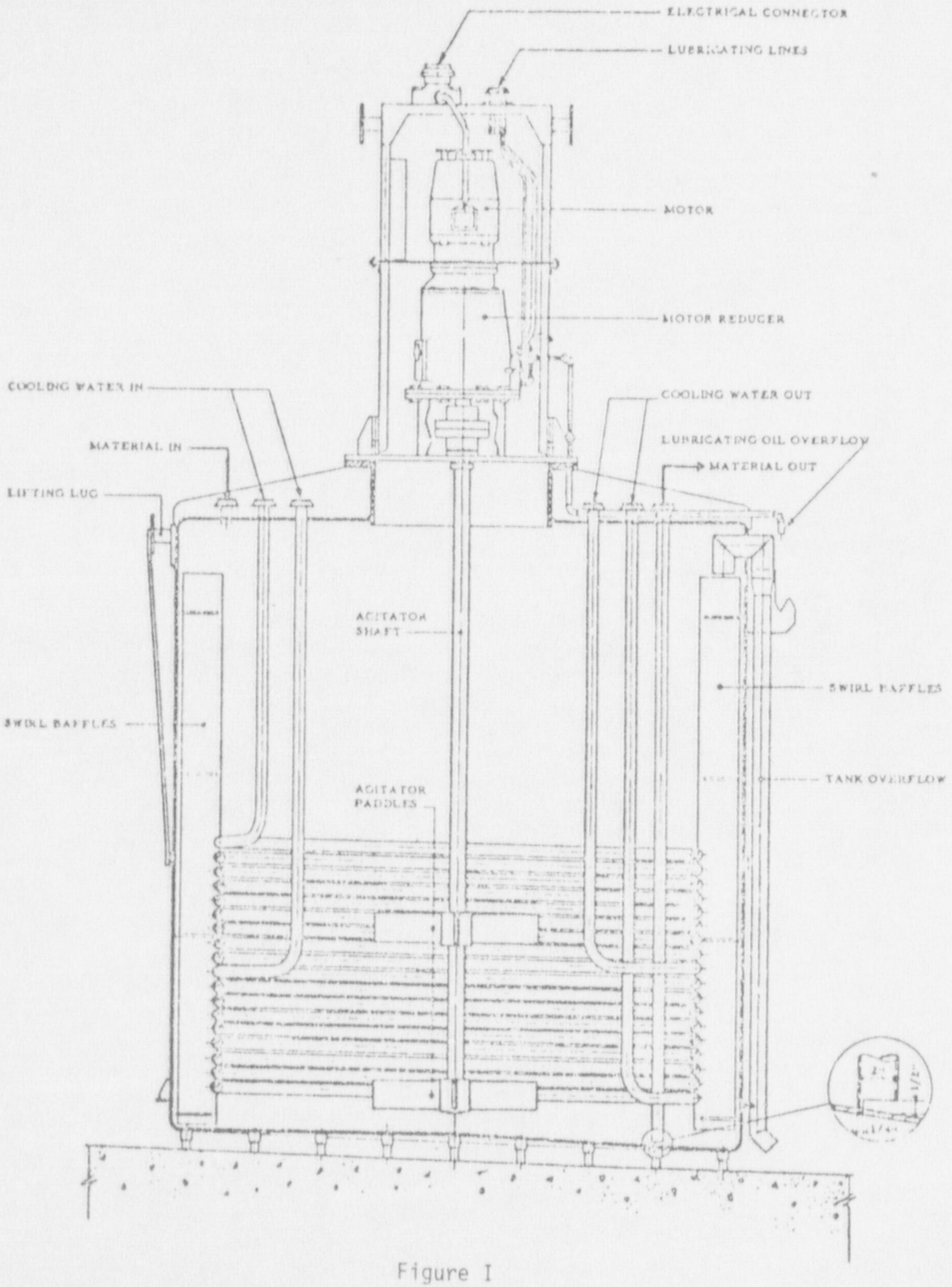
ELECTRICAL CONNECTOR
LUBRICATING LINES
MOTOR
MOTOR REDUCER
COOLING WATER IN
COOLING WATER OUT
MATERIAL IN
LUBRICATING OIL OVERFLOW
MATERIAL OUT
LIFTING LUG
AGITATOR SHAFT
SWIRL BAFFLES
SWIRL BAFFLES
TANK OVERFLOW
AGITATOR PADDLES

Figure I

B. Simulated Process Tank Study

A test setup has been completed for determining the accuracy of the level measurement systems in accountability tanks. This test setup is sketched in Figure 2. The operation of the system is based upon the calibrated relationship between liquid quantity in the tank and the pressure readout system. This system is potentially considered to be affected by such factors as those listed on Table 9. The first ten items will be assessed using water as the working fluid when ambient temperatures prevail. The effects of liquid density and temperature variations will then be examined.

The test setup is pictured in Figure 3. Here small diameter plexiglas tubes simulate the tank being calibrated. Additional tanks of larger diameter will be tested to determine diametral effects. The setup shown is equipped with standard bubbler tube configuration whose position relative to the tank botton, is controlled by means of a precise stepping motor unit visible at the top of the photo. The bubble formation and rise dynamics and the resultant effects on water surface motions at different air flow rates and liquid depths can be very clearly observed or photographed through the clear tube walls. As the bubble formation and "break-off" phenomena reverberate back up the bubbler tube, the pressure signatures can be monitored via fast response pressure transducers shown in the instrument rack. Instantaneous or averaged pressure versus time traces can be analyzed or plotted via the instrumentation rack shown at the left of the photo.

Typical pressure traces are shown in Figures 4 and 5. These figures show the variations in the pressure in the bubbler tube line that occur over the bubble formation and break-off processes. The period of the bubble cycle is noted to vary with depth when the air flow rate is kept constant. It is also noted that the salient features in the pressure trace are reproducible in the two figures. After the initial decrease in pressure that follows bubble break-off from the tip of the tube, a large pressure spike is observed in the trace. This is thought, presently, to be due to the compression effects of the liquid rising in the bubbler tube after break-off. Subsequent pressure variations are compression and rarefaction waves in the air column that decay in some fashion with time. After this decay, the pressure is found to rise slowly with bubble formation followed by the next break-off. Present efforts are focused on this pressure signal - determining its average value and how the signal and its average are altered by the effects listed on Figure A2.

Investigators for this portion of the task are B. Robertson and A. Gaigalas (visiting professor).

ACCURACY OF PROCESS TANK CALIBRATION

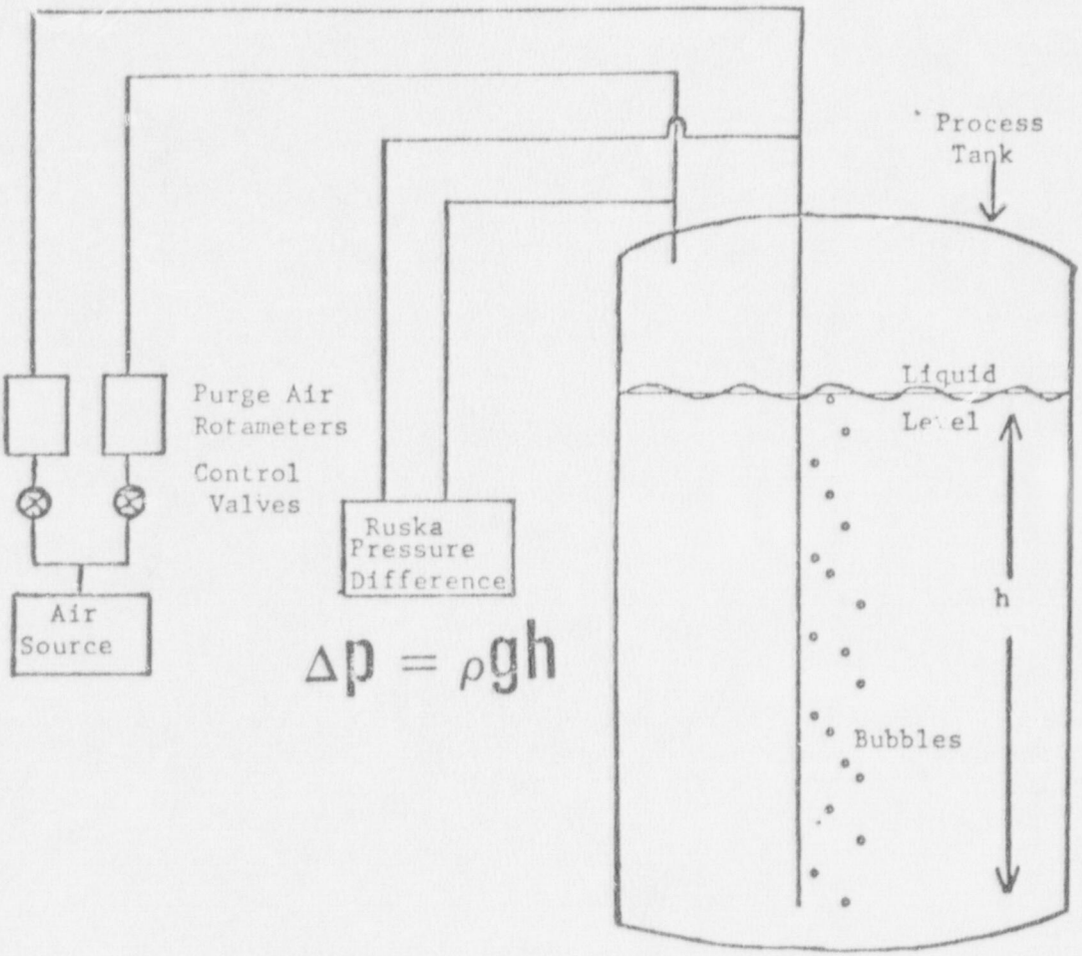

Figure 2

LIQUID LEVEL MEASUREMENT

EFFECT ON ACCURACY DUE TO:

- TANK DIAMETER
- TUBE DIAMETER
- CLOGGING OF TUBE
- OBSTRUCTIONS NEAR TUBE END
- SURFACE WAVES
- FILLING JET
- AIR LINE CONFIGURATION
- AIR FLOW RATE
- SPARGE AIR FLOW
- LIQUID LEVEL
- LIQUID DENSITY
- TEMPERATURE

Table 9

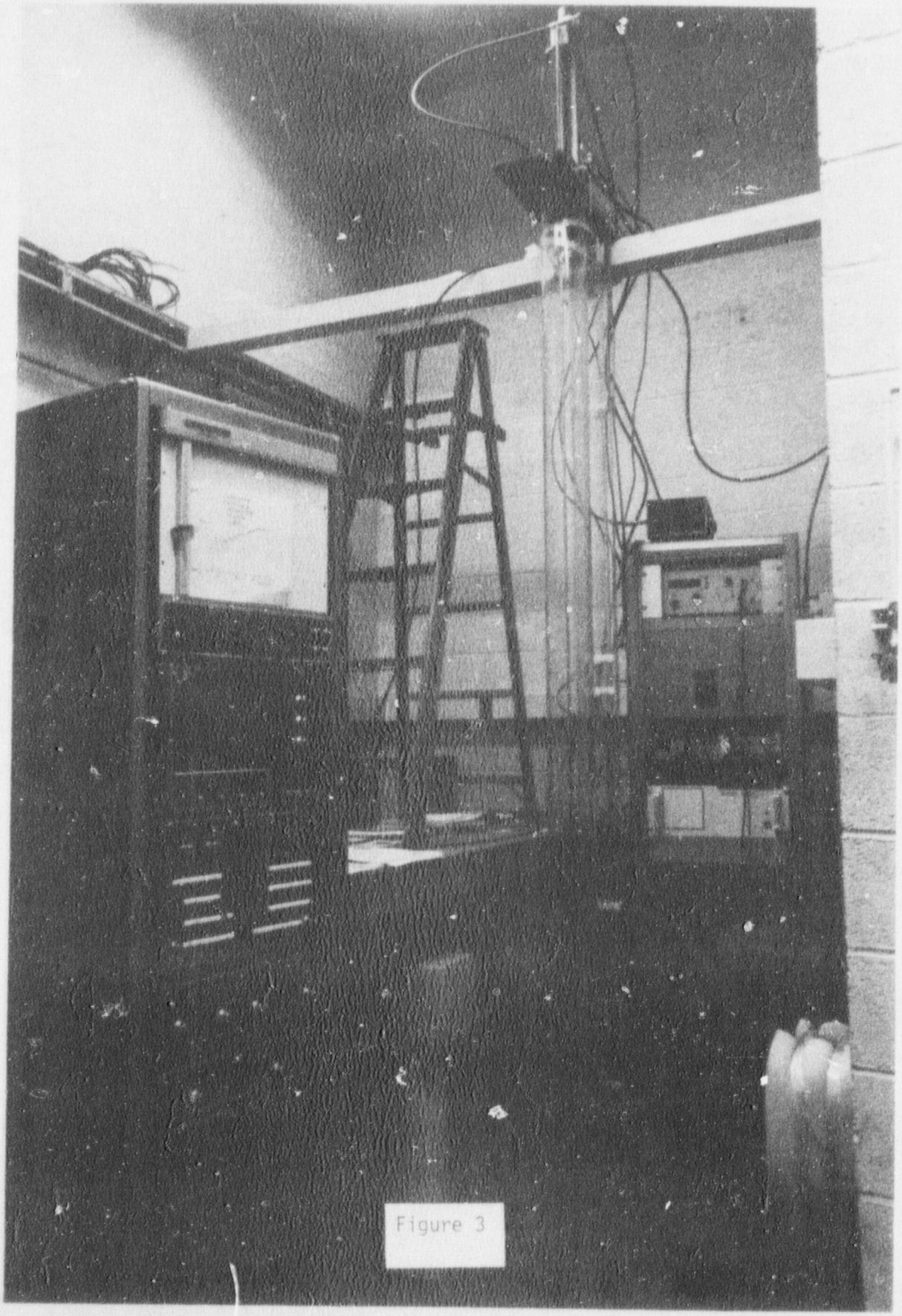

Figure 3

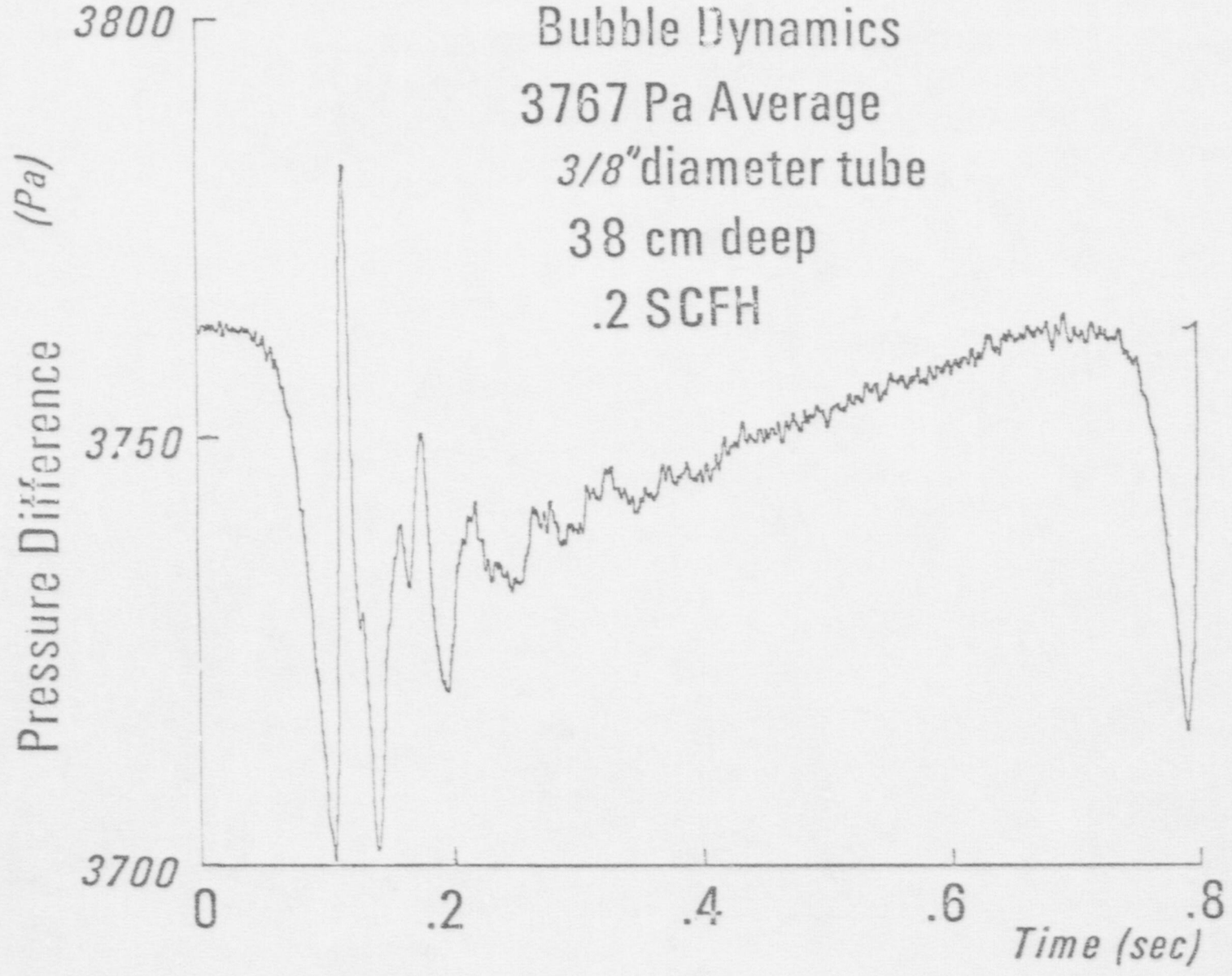
Bubble Dynamics
3767 Pa Average
3/8" diameter tube
38 cm deep
.2 SCFH
3800
3750
3700
Pressure Difference (Pa)
0
.2
.4
.6
.8
Time (sec)

Figure 4

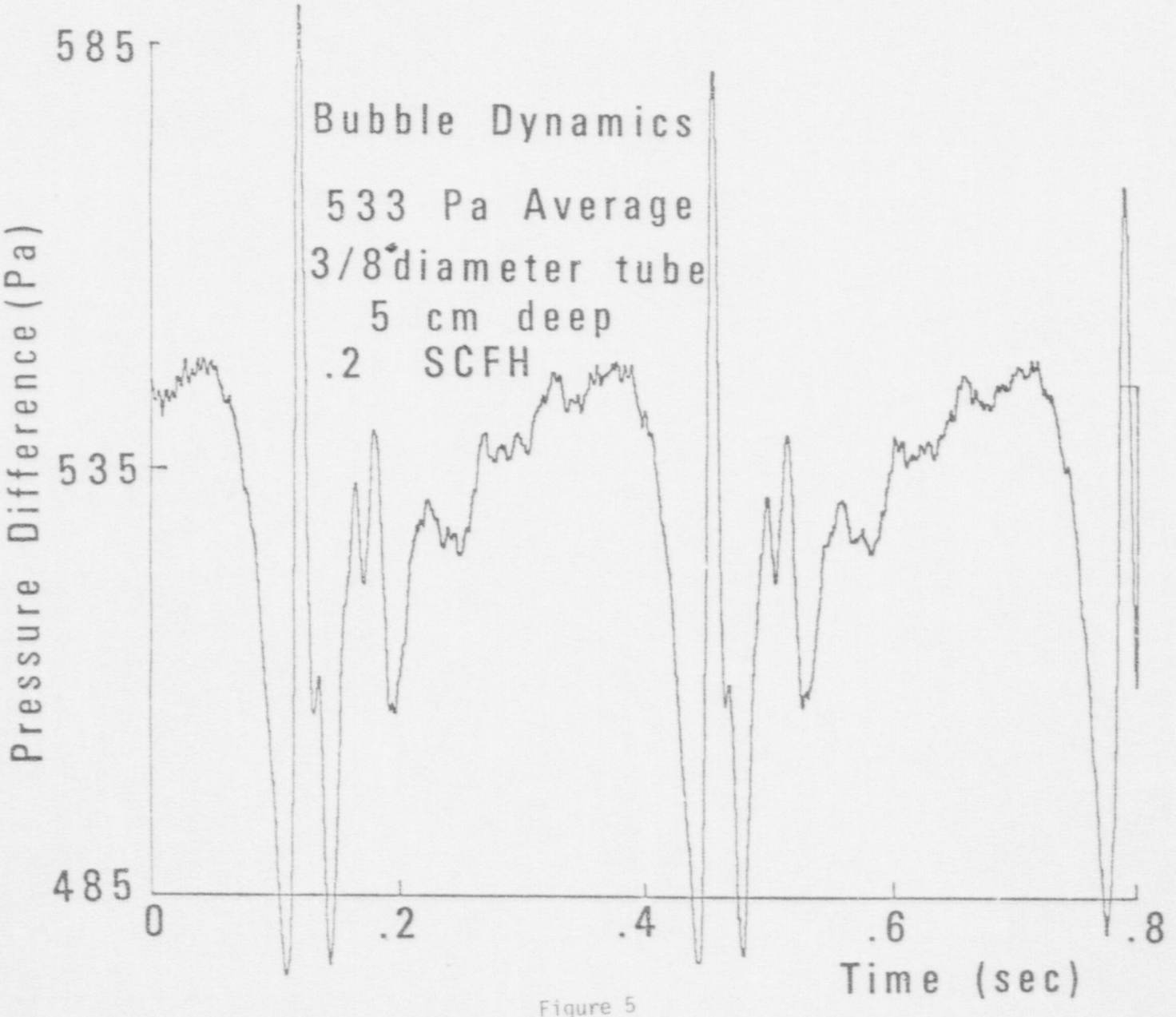
585
535
485
Pressure Difference (Pa)
Bubble Dynamics
533 Pa Average
3/8" diameter tube
5 cm deep
.2 SCFH
0
.2
.4
.6
.8
Time (sec)

Figure 5

C. Test Air-Servoed Weigh Beam

The pilot program involving interchanges of large UF_6 cylinders between three plants is nearing completion. A paper describing this system for safeguarding UF_6 shipments has been accepted for presentation at the forthcoming IAEA Conference to be held in the Fall. The project to demonstrate the operation of a MAP program in this area, initially run by NBS, is in the start-up stage.

We continue to await delivery of the weigh beam which we will investigate for applicability for retrofit of installed multiple lever scales. At this time, we are expecting delivery in ten to twelve weeks. However, this date may slip as the company appears to be undergoing severe reorganizational changes caused by expansion of facilities into the U.S. Since this is the only supplier, we will have to be patient.

Investigators for this portion of the task are P. Pontius and D. Mitchell.

D. Assemble and Test Prototype Dynamic Volume Tank Calibrator

The operational design for the prototype calibrator is sketched as shown in Figure D1. The system, which is computer operated, uses city water as supplied by faucets through house water lines. Accurately determined volumetric quantities of water are supplied to the tank under calibration and the corresponding pressure measurements are recorded from the tank's liquid level measurement system. When the tank is filled or calibrated to a predetermined level, the computer terminates the process and provides a permanent record on a floppy disk. The result would be a table or graph of the liquid volume versus pressure readout value of the liquid level measurement system. By preselecting the approximate flow rate of city water, the time of calibration can, within limits, be predetermined. For large tanks, this time is envisioned to be no more than 30 hours.

The prototype calibrator is now assembled and is shown in Figure 6. The desktop computer is mounted in the rack below a series of counters which monitor the tandemly piped turbine meters which are the heart of the system. To the right of the rack mounted system is the volume standard against which the initial turbine meters are being calibrated. Figure 8 illustrates the turbine meter characteristic curves that are expected from our calibration tests. Given that these results are found as expected, the completion of the circuitry required for inhouse testing of the entire unit will then enable a simulated test on large volume tanks at NBS.

Investigators for this portion of the task are B. Robertson and P. Baumgartner.

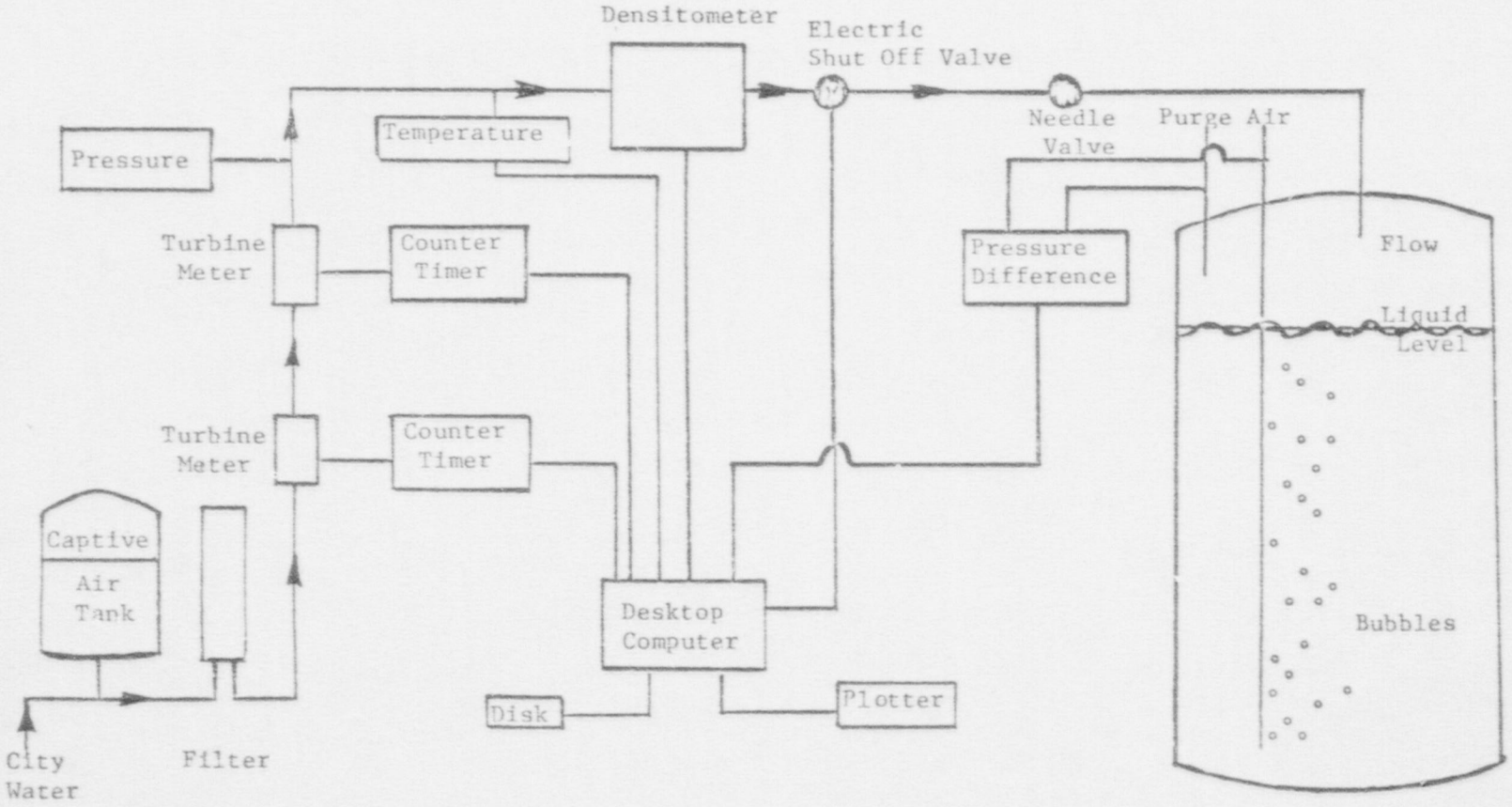
Densitometer
Electric
Shut Off Valve
Needle
Valve
Purge Air
Pressure
Temperature
Turbine
Meter
Counter
Timer
Pressure
Difference
Flow
Liquid
Level
Turbine
Meter
Counter
Timer
Captive
Air
Tank
Desktop
Computer
Bubbles
Disk
Plotter
City
Water
Filter

Figure 6

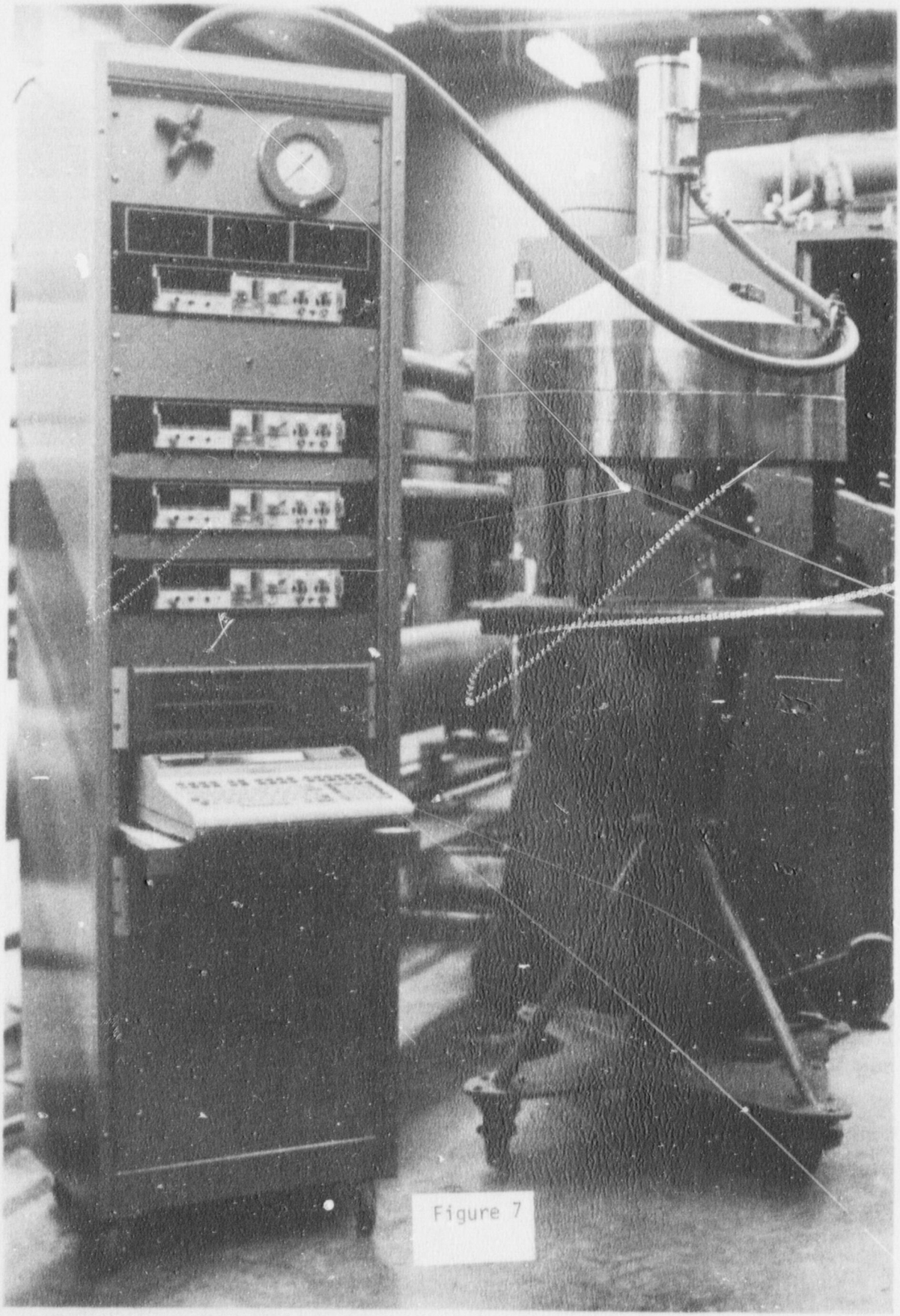

Figure 7

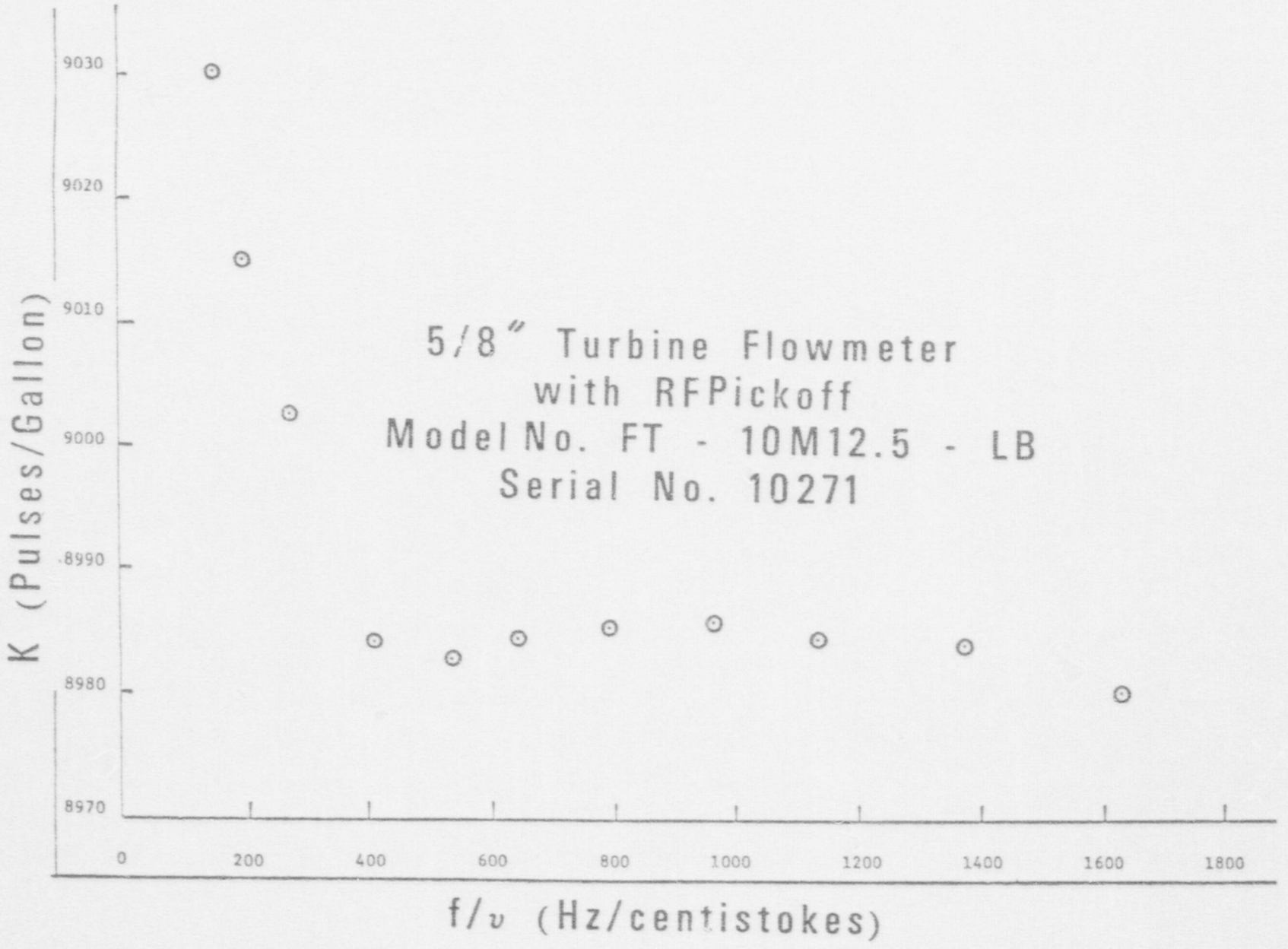
5/8" Turbine Flowmeter
with RFPickoff
Model No. FT - 10M12.5 - LB
Serial No. 10271
K (Pulses/Gallon)
9030
9020
9010
9000
8990
8980
8970
0
200
400
600
800
1000
1200
1400
1600
1800
f/ν (Hz/centistokes)

Figure 8

E. Mobile Flow Standard

The design for the portable flow standard has been completed. The components have been obtained and the bidding process for fabrication has commenced. Bid packages were sent out on June 19. We expect an opening date in early July with the contract let by July 12. It is expected that the fabrication costs (paid by NBS) were initially underestimated by $7,000 to $10,000. Completion may require additional NBS funds at this level.

The investigator for this portion of the task is J. Whetstone.

F. Portable Weighing Device

We have obtained the prototype equipment for the portable weighing device based on load cells. At this time, the equipment is being tested to determine which parts are useful for further development. Servo designs have been started for control of the constant loading mechanism. Preliminary data using the NBS dead weight machines show that the window for the constant load may be as great as 1,000 lbs out of 30,000 within which the constancy of the load cell's indication which corresponds to an uncertainty level in the 1 to 10 pound region.

Investigators for this portion of the task are P. Pontius and D. Mitchell.

G. Pressure Transducer Calibration

In an additional effort to improve liquid volume measurements in tanks, we are moving forward with a pressure transducer calibration service. This service has two parts. We are evaluating three pressure transducers submitted by Rockwell International from their Rocky Flats operations. These units are being evaluated according to the NBS Pressure Characterization Schedule shown in Table 10. Secondly, we are evaluating eight 0-15 PSIA transducers in order to investigate possible transfer standards for use in a Measurement Assurance Program (MAP).

Work has proceeded apace on the evaluation of the three Rockwell units and we have now completed 27 of the 35 tests. There have been no failures among the units despite rapid pressure cycling and extreme temperatures. One fundamental characteristic of the transducers was discovered during the first calibration (test 6 on the Schedule), and that is that the units will not go above 3 PSI even though they are rated at 3.7 PSI. At pressures above 3 PSI, the transducers are saturated and do not respond to changes in pressure. Personnel at Rocky Flats have been notified.

The second part of this project involves an evaluation of pressure transducers which could provide suitable transfer standards for use in a MAP program. Eight 0-15 PSIA units have been looked at and the Ruska DDR6000 appears to be the most stable and reproducible. Consequently, a unit has been ordered for further study.

A small portion of the vast amount of data collected from the Rockwell units has been analyzed. The analysis is expected to be well under way by the end of the next quarter.

Investigators for this portion of the task are V. Bean and Sherry Wood.

Table 10

NBS Pressure Transducer Characterization Schedule

The parameters to be investigated are listed below with the numbers in parenthesis referring to specific tests that apply.

I. Warm up (1)

II. Zero drift (3,15,18,30)

III. Supply voltage dependence (4,5)

IV. Calibration (6,7,14,17,31,33)

V. Precision (6,7,14,17,31,33)

VI. Pressure hysteresis (6,7,14,17,31,33)

VII. Short term stability, 10's of hours (6,7)

VIII. Long term stability, 7 months. (4,33)

IX. Zero drift on a function of temperature (21)

X. Temperature hysteresis (21)

XI. Sensitivity shift as a function of temperature (22,23,24,25,26)

XII. Full scale drift (9,11,19)

XIII. Relaxation effects (10,12,20)

XIV. Effects due to pressure cycling (13,14,15,16,17,18,19,20)

XV. Pressure fluid dependence (8)

XVI. Attitude dependence (34,35)

XVII. Effects due to storage/transportation temperature cycle (27,28,29)

XVIII. Composite calibration equation including temperature effects. (6,7, 14,17,22,23,24,25,26,31,33)

TRANSDUCER TEST SCHEDULE

1. Observe zero drift for a 3 day period taking readings after turn on each 500 seconds for the first 20,000 seconds and each 10,000 seconds thereafter. Leak tight absolute transducers will be evacuated to a pressure of 0.1 Pa or less.

2. Zero the transducers as required. Zero adjustments will not be made again until just before the final calibration.

3. Observe zero drift every 10,000 seconds for 1 week.

4. Decrease the supply voltage to 10% below normal and calibrate against a piston gage (estimated 33 ppm accuracy, 2 ppm precision) at 0, 20, 40, 60, 80, 100% full scale with only increasing pressure allowing no overshoot. Return voltage to normal.

5. Increase the supply voltage to 10% above normal, calibrate at 0, 20, 40, 50, 80, 100% full scale with increasing pressure and no overshoot. Return voltage to normal.

6. Calibrate at room temperature with increasing pressure at 0,10,20,30,40,50,60,70,80,90,100% full scale and 100,95,85,75, 65,55,45,35,23,15,5,0% full scale with decreasing pressure allowing no overshoot.

7. Calibrate again

8. If appropriate, calibrate using He as the pressure fluid.

9. Pressurize to full scale, hold the pressure for 3 weeks, compare full scale reading with piston gage each working day.

10. Reduce pressure to 0, observe zero drift for 1 week.

11. Repeat 9

12. Repeat 10

13. Pressure cycle from 0 to 90% of full scale 3450 times with interruptions each 690 cycles to observe zero drift for an hour at 500 second intervals.

14. Calibrate after 24 hours of zero drift measurements after the end of cycling.

15. Observe zero drift for 1 week.

16. Pressure cycle from 0 to 90% of full scale 6900 additional times with interruptions each 690 cycles to obersve zero drift for an hour at 500 second intervals.

17. Calibrate after 24 hours of zero drift measurements after the end of cycling.

18. Observe zero drift for 1 week.

19. Pressurize to full scale, hold for 3 weeks, compare reading with the piston gage each working day.

20. Reduce pressure to zero, observe zero drift 1 week.

21. While making zero drift measurements, increase the transducer temperature at 40 °C, decrease to 0 °C, then increase to 23 °C at as rapid a rate as the environmental chamber allows (several hours).

22. Check the calibration at 0, 20, 40, 60, 80, 100% full scale using only increasing pressure while at 40 °C.

23. Lower the temperature to nominal 31.5 °C, check the calibration at 0, 20, 40, 60, 80, 100% full scale using only increasing pressure.

24. Lower the temperature to nominal 11.5 °C, check the calibration at 0, 20, 40, 60, 80, 100% full scale using only increasing pressure.

25. Lower the temperature to nominal 0 °C. Check the calibration at 0, 20, 40, 60, 80, 100% full scale using only increasing pressure.

26. Return to room temperature, check the calibration at 0, 20, 40, 60, 80, 100% full scale using only increasing pressure.

27. Turn off the power to the transducer, increase the temperature to 50 °C, hold for 4 hours, decrease the temperature to -40 °C, hold for 4 hours, return to room temperature.

28. Turn on power, allow 1 week warm up while observing zero drift at 10,000 second intervals.

29. Check the calibration at 0, 20, 40, 60, 80, 100% full scale using only increasing pressures.

30. Observe zero drift for 4 weeks.

31. Calibrate.

32. Adjust the zero.

33. Calibrate again.

34. If appropriate, tilt the transducer by 1,2,3,4,5,10,15,30,60,90° about two orthogonal horizontal axes while observing zero drift.

35. Repeat 34 at full scale pressure.

NRC FORM 335 (7-77) U.S. NUCLEAR REGULATORY COMMISSION

BIBLIOGRAPHIC DATA SHEET

1. REPORT NUMBER (Assigned by DDC): NUREG/CR--0289

4. TITLE AND SUBTITLE (Add Volume No., if appropriate): Measurements and Standards for Nuclear Materials Safeguards Quarterly Report February 1, 1978 - April 30, 1978

2. (Leave blank)

3. RECIPIENT'S ACCESSION NO.

7. AUTHOR(S): H. T. Yolken, F. E. Jones

5. DATE REPORT COMPLETED: MONTH July, YEAR 1978

9. PERFORMING ORGANIZATION NAME AND MAILING ADDRESS (Include Zip Code):
U.S. Department of Commerce
National Bureau of Standards
Physics Building
Washington, D.C. 20234

DATE REPORT ISSUED: MONTH July, YEAR 1978

6. (Leave blank)

8. (Leave blank)

12. SPONSORING ORGANIZATION NAME AND MAILING ADDRESS (Include Zip Code):
Division of Safeguards, Fuel Cycle & Environmental Research
Office of Nuclear Regulatory Research
U.S. Nuclear Regulatory Commission
Washington, D.C. 20555

10. PROJECT/TASK/WORK UNIT NO.: B6016

11. CONTRACT NO.: AT(49-25)-9009

13. TYPE OF REPORT: Technical

PERIOD COVERED (Inclusive dates): February 1, 1978 - April 30, 1978

15. SUPPLEMENTARY NOTES

14. (Leave blank)

16. ABSTRACT (200 words or less)

This report is a review of the third quarter, (February 1, 1978 thru April 30, 1978), of a long-term NBS program sponsored by NRC to up-grade national measurements and standards capability for nuclear materials safeguards.

The overall approach that NBS is utilizing to provide for development and dissemination of a consistent set of national measurement standards for nuclear materials safeguards is presented. It should be stressed that a great deal of work needs to be done to provide the standardization base for alternate fuel cycles. Many materials such as thorium, Uranium 233, and plutonium or mixed oxides "spiked" with fission products might well be found in future alternate fuel cycles. The NBS program is aimed at providing both the standards for today's needs and the standards for future fuel cycles.

A summary of the progress for each of the five tasks in the project is given.

17. KEY WORDS AND DOCUMENT ANALYSIS

17a. DESCRIPTORS

17b. IDENTIFIERS/OPEN-ENDED TERMS

18. AVAILABILITY STATEMENT

19. SECURITY CLASS (This report): UNCLASSIFIED

21. NO. OF PAGES: 36

20. SECURITY CLASS (This page): UNCLASSIFIED

22. PRICE: $

NRC FORM 335 (7-77)

UNITED STATES
NUCLEAR REGULATORY COMMISSION
WASHINGTON, D. C. 20555

OFFICIAL BUSINESS
PENALTY FOR PRIVATE USE, $300

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