Meetings

Carbon-14 and Tritium Dating

Since 1954 workers in radiocarbon dating have met six times for international gatherings devoted solely to considering the assumptions, techniques, and applications of the age determination method pioneered by Willard Libby. For the sixth and most recent international meeting over 145 conferees met in Pullman, Washington, from 7-11 June 1965. About 48 foreign scientists attended and presented slightly more than half of the papers. For the first time, papers on the measurement of environmental tritium were included in the conference because of a certain overlap with radiocarbon dating in both technique and application. Libby served as honorary chairman and delivered an address entitled "Radiocarbon and tritium in retrospect and prospect."

Special addresses were given by Thomas A. Rafter (New Zealand Institute of Nuclear Sciences) and Fred Johnson (R. F. Peabody Foundation for Archeology). One of the first workers to begin natural radiocarbon measurement following Libby's initial publications, Rafter spoke on "Problems in the establishment of a carbon-14 and tritium laboratory." Johnson spoke to the group on "The impact of radiocarbon dating on archeology." Few people have such insight to this dramatic effect as Johnson, who was on the original committee of five called by Libby to secure known-age samples for initially testing the technique of carbon-14 dating.

Archeologic Applications

Since no group of scientists has used radiocarbon dating more than archeologists, three sessions were held in which the archeologic contributions, problems, and limitations of C^{14} dating were outlined. Griffin (Michigan) focused his remarks on samples that came from eastern United States. He noted the absence of satisfactory dating of many Archaic complexes and a conflict between archeological interpretations and radiocarbon dates for northern and southern Hopewellian cultures. He urged greater care in selection of radiocarbon samples in the future. Neuman (Smithsonian Institution) reported on archeology of the American Great Plains based on 214 dates from 121 sites. The dates show that the sites were occupied by hunters and gatherers in the interval from 9000 to 100 B.C. The initial ceramic cultures date from around A.D. 50 to 700, and agricultural settlements along the Missouri River and its tributaries fall within the interval A.D. 700 to 1800.

In his report on the American Southwest, Jelinek (Michigan) described an attempt to correlate C¹⁴ dating and palynology for the Cochise sequence in southeastern Arizona. Jelinek noted the lack of an adequate number of dates when he pointed up gaps in the dating record from 8900 to 7500 B.C. and from 4900 to 3300 B.C. In addition, dates for ceramic cultures are absent. Illustrating the dearth of C¹⁴ ages is the fact that only 12 sites account for 60 percent of radiocarbon ages in the southwest.

Haynes (Arizona) presented conclusions from a study of radiocarbon dates on early man sites in the United States. Classifying the available dates into 500-year intervals from about 11,-500 years ago (Clovis points), Haynes traced the diversification of projectile point types over a 3500-year period. In fact, some points appear to be usable as index fossils. Such a use suggests that some C¹⁴ dates from caves should be questioned. From his critical review, Haynes found that no reported C14 dates older than 10,000 B.C. can be positively related to early man in the United States. He was impressed with the stratigraphic consistency of certain artifact types, fossils, and C^{14} dates.

Borden (British Columbia) told of work he has done at three sites in the Lower Fraser Canyon near Yale, British Columbia, where a series of seven cultural phases dates from the 8th millennium B.C. almost to the present. He was able to relate human occupation to late Glacial geologic activity. Campbell (New Mexico) focused attention on the C¹⁴ chronology of Arctic sites as it relates to chronologies based on other dating methods. In a paper on Peruvian archeology, Rowe (University of California, Berkeley) described the problem of inconsistent results between C¹⁴ dates made recently and those carried out in the midfifties. The later results move the time scale back around 600 years, suggesting that the earlier values reflect difficulties with the obsolete black-carbon measurement technique.

In discussing dates from southwestern Europe and the Mediterranean Basin, Smith (Toronto) pointed out that the dating of the Mousterian sequence is not clear, and even in southwestern Europe the subdivisions of the Upper Paleolithic are not yet firmly dated, particularly in Spain and Italy. Judging from several C^{14} dates from Libya, North Africa seems to have had a surprisingly early form of Upper Paleolithic, while the lower Nile Valley was also less retarded than is usually thought.

Vértès (Budapest) discussed the evidence for the transition from Middle to Upper Paleolithic in Eastern Europe, where available C¹⁴ dates indicate it occurred somewhat earlier than in Western Europe. However, one cannot be sure the transition truly represents an independent development. Vogel (Groningen) reported on the evidence suggesting that some of the Mousterian and early Upper Paleolithic dates from Western Europe may be too young by several millennia. He also introduced new evidence that the Paudorf oscillation extended from 30,000 to 26,000 B.C. and that the previously accepted Gottweig dates should be modified as well. Bandi (Bern) discussed the Mesolithic of Western Europe, especially of Switzerland, and the evolution of the Epipaleolithic cultures in new environment. His results are based in part on his own work in aligning the cultural stages with late Glacial and early post-Glacial climatic history.

Clark (Cambridge) outlined the important contribution radiocarbon dating has already made to an understanding of the diffusion of agriculture in the Old World. He pointed out that the dates have tended both to confirm the Middle East as the ecologically focal area, and to establish the priority of the Greek lands and the Middle Danube in the earliest spread into Europe. The dates have also greatly expanded the time span of the whole European Neolithic to the point where important chronological subdivisions can now be established.

Waterbolk (Groningen), in describing the Copper Age of southeastern Europe, pointed out that in the light of recent radiocarbon dates we must revise our idea that copper working was earlier in the Iberian Peninsula than in Southeast Europe.

C. A. Moberg (Göteborg) emphasized the problems involved in dating the many scattered, unstratified sites of food-collecting peoples in Scandinavia during the last 5000 years. There is also great difficulty in correlating the chronology of northern Scandinavia with the better known southern Scandinavian sequence.

Agrawal (Bombay) presented a C^{14} chronology for the Harappa culture, including its terminal phases, and for the first wave of Aryan-speakers, represented by the Banas culture. The "Dark Ages" of India between about 1500 to 500 B.C. are now being filled in as Painted Gray-Ware sites are dated by radiocarbon.

A general session is admittedly one designed to catch all papers not fitting into established categories. However, in a session devoted to archeology in general there seemed to be the common note of watchfulness in relaing C14 dates to acheologic problems. Stuckenrath (Pennsylvania) and Davis (Texas) sounded similar notes in their attempts to point up fundamental problems which may invalidate C^{14} dates. They both emphasized the need for close cooperation between the collector and the laboratory personnel who make the radiometric analysis. In the connected fields of soil science and stratigraphic archeology, Dimbleby (London) described the potentialities and problems of radiocarbon dating. He suggested three specific applications: (i) turnover times in active soils, particularly in podzol B horizons; (ii) time of change in soil character as a consequence of human or natural influences; and (iii) the approximate age of buried paleosols.

Shutler (Carson City) mentioned problems unique to the tropics where heavy rainfall may potentially cause contamination of samples. Where there are several internally consistent dates from a single site, there is reason for confidence. Shutler reported ages of earliest settlement in several Pacific islands, among which Sarawak showed the oldest age at 39,000 B.C. and the Philippines next at 20,000 B.C. Based on his series of ages, he inferred two possible migration routes outward from mainland Asia to the various Pacific islands.

Of particular interest was a paper by Oakley (British Museum) concerning C14 dating of fossil hominids. Oakley emphasized that there are really four distinct ways of determining the absolute age of fossil human remains: (i) from the material itself, (ii) from associated material, (iii) from correlation with similar deposits dated absolutely, and (iv) inference on a largely theoretical basis. It was Oakley's contention that our knowledge of the age and relationship of late Pleistocene hominids would be improved by a general recognition of these four methods of absolute dating.

Geologic Applications

Dreimanis and Vogel (Western Ontario and Groningen) presented new dates and interpretations defining Wisconsin glacial fluctuations north of Lake Erie. Gonzales, van der Hammen, and Flint (Colombia, South America; Leiden; and Yale) described a C14-calibrated pollen sequence found in lake sediments related to mountain glacier activity in the high Andes of Colombia. Their results support the view that major climatic events there were synchronous over the past 12,000 years with those in mid- and highlatitude North America and Europe. A similar time interval was the concern of Fryxell (Washington State) who integrated 50 C14 dates into a post-glacial chronology of the Columbia Plateau; two well-dated ashfalls (Mt. Mazana at 6600 years ago and Glacier Peak at 12,000 years ago) were especially useful as stratigraphic markers.

Berger, Ting, and Libby (University of California, Los Angeles) reported on radiocarbon dated materials—primarily coprolites—which are associated with pollen in caves of southwestern United States.

The oölithic sands of the Bahama Banks were the topic of Martin and Ginsburg (Shell Development Company). Chemical separation of these concentric spheroids into inner and outer fractions was followed by dating, results of which were consistent with radial growth. Since no apparent age older than 2700 years was found, a relatively rapid growth rate is indicated.

Today, many laboratories are measuring natural samples with heavy concentrations of C^{14} and H^3 derived from nuclear bomb explosions. Although the dating aspect is not involved, there is much to be learned of mixing rates and turnover patterns in the atmosphere and ocean. This occurs partly because both radiocarbon and tritium exist in the atmosphere as gases (CO₂ and H₂O) in contrast to particulate fallout.

Fergusson (University of California, Los Angeles, and Johnston Laboratories) presented recent stratospheric measurements for the Northern Hemisphere which showed maximum C14 levels ten times the pre-bomb level at the earth's surface. A decrease in radiocarbon concentration occurs with decrease in latitude and altitude, although strong stratification is found in some sets of measurements. Additional stratospheric data for the Southern Hemisphere showed the familiar 18 months' lag behind Northern Hemisphere values. For the troposphere, Lal (India) presented similar data measured in his country. Besides discussing the nature and time scale of north-south mixing in the troposphere. he included data on strontium-90 in rain in order to obtain an insight into how fallout is washed from the atmosphere.

An original method of determining the source of water in the eye of a hurricane was described by Ostlund (Miami). Noting that rain accompanying 1964 hurricanes seemed depleted in tritium, he arranged for aircraft sampling of water droplets suspended at various points within hurricane Hilda. A more extensive sampling program is planned for the 1965 hurricane season during which the extent of vortex interaction with the stratosphere will be investigated.

Recent surface ocean measurements of both tritium and radiocarbon were presented by La Jolla scientists. Houtermans and Suess reported that 4 years of bimonthly sampling from 12 stations in the Pacific Ocean show that tritium concentrations are rising and that the values in the Northern Hemisphere exceed those in the Southern Hemisphere. Radiocarbon measurements were given by Bien, Rakestraw, and Suess who found evidence of a steady rise in concentration since 1954. The rate at which C14 produced by bomb explosions is taken up and mixed into the water masses of the deep ocean gives information on CO2 exchange between atmosphere and ocean. Both radioisotopes provide evidence on rates at which surface water mixes downward through the thermocline.

Münnich reviewed the mixing processes in and between the atmosphere, ocean, and groundwater reservoir. He noted the inadequacy of simple box models to explain data that are becoming increasingly refined. He then described convenient ways to handle more complex models. His illustrations concerned vertical mixing within the ocean and the atmosphere (where both C^{14} and H^3 are useful) and soil water transport (where H^3 is most useful).

Interest in water supply problems attracted many to the session on hydrological applications of C^{14} and H³. In fact, a dozen of the conferees gathered on two separate occasions to continue discussions begun in the session itself. Owing to the difference in half-lives (5730 years for C^{14} and 12.26 years for H³), both slow and fast water movements may be studied with these two radioisotopes.

Three areas well known hydrologically were topics for papers describing the use of radiocarbon in groundwater, Rubin, Hanshaw, and Back (U.S. Geological Survey) found increasing C¹⁴ ages down the piezometric surface of the Ocala, Florida, limestone aquifer; their calculated rate of movement was 25 feet per year, in excellent agreement with estimates based on permeability methods. Similar agreement was found by Bennett (Arizona) for an artesian aquifer in Arizona, and studies are being extended to the Tucson Basin. Geyh and Wendt (Hannover) added stable carbon isotope measurements to radiocarbon data in order to study exchange processes between dissolved CO₂ and calcite within the aquifer.

Unlike the situation with radiocarbon, bomb-produced tritium is today

so abundant as to dwarf natural concentrations of the isotope. Hence, the use of tritium in hydrology requires many measurements today in order to establish the base lines necessary for future hydrological conclusions. The conference was made aware that there is a great amount of data now being accumulated routinely in a number of areas. For example, Thatcher and Payne (International Atomic Energy Agency, Vienna) and Stewart (U.S. Geological Survey) presented results that have been gathered by their organizations. Both seasonal and yearby-year tritium variations were demonstrated.

For semiarid regions of the Mediterranean, Gat (Israel) described some of the problems and techniques involved in using bomb-produced tritium as a tracer to discover turnover times and mixing patterns of lakes and groundwater. The problems include analysis, sampling, isotopic fractionation, and isotopic exchange.

Fundamental Studies

Throughout the conference emphasis was placed on the fact that laboratories do not measure ages, they measure sample activities. The connection between activity and age is made through a set of assumptions. However, in relating tree rings and carbon-14 dating there is little need to emphasize the above point, because one of the main assumptions of C^{14} dating is that the atmospheric radiocarbon level has held steady over the age-range to which the method applies. As indicators of atmospheric C¹⁴ levels in the past, tree rings have been selected on the basis that the carbon in a given ring reflects the atmospheric level when it was formed, provided correction has been made for isotopic fractionation through use of the stable isotope ratio (C^{13} : C^{12}).

Dendrochronologist Bannister (Arizona) emphasized the danger of universally assuming that a given tree ring can be dated merely by counting radially inward from the date of tree cutting. He outlined the laborious effort involved in detecting missing annual rings or multiple rings within a single year. The other participants reported on tree ring analyses; many authors suggested ways to explain deviations from the atmospheric uniformity assumed in C^{14} dating.

Schell, Fairhall, and Harp (Wash-

ington) analyzed the cellulose from Sequoia gigantea rings back to 700 B.C. Their results show a deviation that may be explained in one of two ways. Either the half-life of radiocarbon is 5833 ± 127 , slightly higher than the presently accepted value, or ocean temperature has linearly changed and hence gradually altered the ocean-atmosphere equilibrium relations and in turn the ratio of C¹⁴ to C¹² in the air.

Kigoshi (Tokyo) reported on the analysis of an 1800-year-old tree from Yaku Island in southern Japan. His results show a continuous decrease totaling about $2\frac{1}{2}$ percent during the past 1800 years. For 1500 years of this period Kigoshi suggests correlation with measured variations in the earth's magnetic intensity as preserved in bricks historically dated. Such a magnetic variation would cause a variation in the cosmic ray flux striking the earth's atmosphere and in turn would alter the rate of C¹⁴ production.

Damon (Arizona) presented results on some of the oldest rings dated so far. He found a rise of 0.4 percent every 100 years in the atmospheric C¹⁴ level from 500 B.C. back to 2400 B.C.; the latter date is represented by rings from bristlecone pine. Egyptian archeological samples of the 3rd millennium B.C. suggest the same high atmospheric C14 level. In an effort to extend the sampling period still further, the University of Arizona Dendrochronological Laboratory has succeeded in attaching the outer rings of a long-dead bristlecone pine to the earliest part of the Southwest chronology, thereby making available an inner ring some 5700 years old. Damon reported that the atmospheric C¹⁴ level indicated by this ring is 13 percent above that accepted as the standard used in age calculations and is equivalent to an age error of approximately 1000 years. A possible climatic element is Damon's suggested cause of atmospheric variation, but he presented no specific mechanisms.

Dyck (Geological Survey of Canada) told about Douglas fir rings spanning the past 1100 years. His results confirm previously published work. To explain the observed variations (which fall within a total range of $4\frac{1}{2}$ percent), Dyck suggested that photosynthetic rates vary enough to account for short-term changes, while redistribution of carbon between the hydrosphere and the biosphere explains long-term changes. Both phenomena presumably depend on climatic variations of temperature.

Stuiver (Yale) and Vogel (Groningen) considered possible geographic variations of contemporaneous rings grown over the past several hundred years. For North America, Stuiver found uniformity regardless of latitude or elevation of the growth environment. Vogel's results for trees from Germany, Argentina, and the United States also agreed, provided corrections were made for isotopic fractionation. Both men measure with statistical errors approximating one-fourth percent.

Based on 150 tree ring measurements, Suess (La Jolla) suggested a correlation between the atmospheric radiocarbon level and sunspot frequency. He therefore postulated that atmospheric C^{14} variations, occurring with a period approximating 100 years, are due to variations in the solar component of the cosmic ray flux. When the time constant is of the order of 1000 years, the C^{14} level of the entire cycle is affected; but so far there is no possible correlation of the existing tree ring data with any relevant geophysical parameter.

The first part of a general session on radiocarbon dating concentrated on problem materials submitted for age dating. Krueger (Geochron Laboratories) presented some encouraging results on the bone collagen remaining after other bone matter is dissolved in cold 0.5 normal hydrochloric acid. Although age diminishes the amount of collagen remaining in bone, some samples older than 40,000 years have yielded enough collagen for dating. Despite carbonate fractions that yield young ages because of contamination by groundwater, collagen fractions have uniformly given what appear to be acceptable ages.

Mortar is another useful material provided accurate dating is shown to be possible. If all of the carbon in mortar is taken from the atmosphere during the period of hardening, accurate dates should be obtainable. But the presence of C^{14} -free limestone in either the sand filler or the slaked lime will make measured ages too old. The standard empirical way to evaluate a given material is to date samples of known age. Such measurements were carried out for mortars by two laboratories and yielded opposite conclu-

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sions. Delibrias and Labeyrie (Gif-sur-Yvette, France) described their work and concluded that radiocarbon dating can be extended to mortars without any modifications required. On the other hand, Stuiver and Smith (Yale) reported that all of the seven mortars they measured showed ages averaging 240 years greater than corresponding historical ages. Hence, mortar must remain a questionable dating material.

Tests of foraminifera derived from deep-sea cores were the subject of a paper by Olsson (Uppsala) who focused on potential contaminants. These she listed as: (i) fine particulate limestone carbonate introduced through sediment reworking, and (ii) atmospheric CO_2 exchanged while a sample is in storage or being processed. Particle size fractionation often isolates the first contaminant, while exchanged surficial CO_2 may be removed by acid leaching or thermal decomposition.

In a paper on groundwater C^{14} ages based on dissolved carbonate analysis, Pearson (Texas) showed that satisfactory correction for the nonatmospheric limestone contribution was possible through use of a stable carbon isotope measurement. Without the correction, measured radiocarbon activities do not correspond to ages determined from hydrologic parameters; with the correction there is good agreement.

The latter part of the general session involved an assortment of topics. Libby (University of California, Los Angeles) pointed up the geophysical insights that might be possible from anomalies in radiocarbon dates-specifically those anomalies that appear to stem from variations in C14 production rates by cosmic rays. He presented evidence that both the solar and terrestrial magnetic moments have remained roughly constant over a time span measurable in tens of thousands of years. Olsson (Uppsala) presented a Fortran computer program that her laboratory technicians may use for C14 age calculations during her absences from the laboratory. And Callow (National Physical Laboratory, England) made a plea for uniformity in the calculation and presentation of C^{14} age results, especially with regard to errors auoted.

The final paper by Thurber, Broecker, and Kaufman (Columbia) provided encouraging evidence that atmospheric uniformity over the past 25,000 years

is approximately true despite the smaller scale variations brought out in the tree ring session. The Columbia scientists determined two distinct ages on a number of marine carbonates—one age based on radiocarbon and the second on the degree of inequilibrium between Th²³⁰ (ionium) and its parent U²³⁴. There was agreement, within the experimental error, over the range 3000 to 25,000 years ago. However, beyond 25,000 years, radiocarbon ages were much younger than corresponding ionium ages—a situation explained as resulting from C¹⁴ contamination.

Measurement Techniques

Since the previous radiocarbon conference, the closest approach to a breakthrough in C¹⁴ techniques is the work of Tamers (Caracas) and of Noakes, Kim, and Stipp (Oak Ridge) in synthesizing benzene for counting by the liquid scintillation method. They have simplified the chemistry and increased the yield beyond 90 percent by using lithium to form the metallic carbide that yields acetylene upon hydrolysis and by polymerizing the acetylene to benzene by vanadium catalysis. Problems of isotopic fractionation, radon contamination, and reproducibility have apparently been solved. With the newer commercial liquid scintillation counters, background and contemporary count rates are such as to give a dating range comparable to the ranges provided by gas counting systems. In addition, automatic sample changing permits frequent measurement of background.

Despite the promise of liquid scintillation counting, gas counting techniques still dominate. Nydal (Trondheim) described a decade of experience with CO_2 ; his techniques of gas purification, background reduction, and counter stability have continually improved through the years. Long (Smithsonian Institution) cited favorable experience with methane prepared by catalytic hydrogenation of CO₂. In this regard, Fairhall (Washington) brought up the difficulty of securing tritium-free hydrogen from commercial suppliers. Sharp (Sharp Laboratories) described his efforts at optimizing counting characteristics in the methane counters produced by his company; he emphasized the solution of electronic problems such as noise and instability. Geyh (Hannover) employs ethane as his counting gas in order to introduce twice the amount of carbon into a liter-atmosphere of sample gas; counting 24 liter-atmospheres of ethane for 6 days allows him to measure, without isotopic enrichment, samples 60,000 years old. Unfortunately a very small amount of sample contamination is very significant in this range and may make statistical calculations of range purely academic. Badly needed for evaluating contamination is a series of samples extending from around 20,000 years, where C^{14} ages should be reliable, back to 75,000 years or beyond.

Finally, Oeschger (Bern) described a very small gas counter having a volume of 40 cm³, designed to analyze CO₂ extracted from glacial ice. Even with so small a detector almost one ton of ice must be melted to yield sufficient CO_2 for radiocarbon measurement.

In the field of natural tritium measurement, it is often necessary to enrich the H³ prior to counting in order to attain adequate sensitivity. Customarily this has been done by water electrolysis, one installation of which was described by Cameron and Payne (International Atomic Energy Agency,

Vienna). Enrichment using thermal diffusion has lately been considered; the conference heard reports on this technique from Sellschop (South Africa) and von Buttlar and Wiik (Darmstadt). Enrichment by use of a gas chromatographic column was described by Smith and Ahktar (Tennessee) but is not as yet applicable to natural levels.

Isotopic enrichment is often unnecessary where bomb-produced tritium is sufficiently abundant. In this case proportional counting has been used without enrichment. Von Buttlar, Wohlfahrt, and Farzine (Darmstadt) generate hydrogen from natural waters and use it to hydrogenate inactive ethylene to ethane, which they count. Lal (Bombay) described a process to produce tritiated methane from water in one stage. His reactor is loaded with sample water, zinc metal, and inactive CO_2 gas; the net reaction is CO_2 + $2H_2O + 4Zn \rightarrow 4ZnO + CH_4$. This same reaction can be used for C14 measurements, in which case the CO_2 is sample-derived and the water is inactive.

The conferees considered the question of the best half-life to use in reporting C14 ages to the journal Radiocarbon. A similar discussion was held during the 1962 Cambridge Conference as a result of three new measurements of half-life (1) that showed the accepted value of 5568 years to be low by 3 percent. Majority opinion in both the Cambridge (2) and Pullman (3) conferences was for retaining the old value for the sake of uniformity in publication while at the same time suggesting a correction factor of 1.03 to be applied for greater accuracy.

One of the highlights for many was the all-day field trip which provided a change of pace in the middle of the conference week. The itinerary included the Palouse hills of Pleistocene loess deposits, the channeled scablands where Pleistocene flood waters have exposed and scoured Tertiary basalt flows of the Columbia Plateau. and the Marmes Rock Shelter at the confluence of the Palouse and Snake Rivers. The latter has been excavated by Washington State University archeologists who have exhumed several human skeletons antedating the Mazama ash fall of 6500 years ago. Guides for the field trip were Richard Daugherty in archeology, Roald Fryxell in geology, and James Crosby in geohydrology.

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Forthcoming Events

December

20-21. Molecular Transport and Rate Phenomena, 32nd annual chemical engineering symp., Stanford Univ., Stanford, Calif. (A. Acrivos, Dept. of Chemical Engineering, Stanford Univ., Stanford, Calif.)

20-21. Nuclear Medicine, 2nd natl. congr., Tel Aviv, Israel. (P. Czerniak, Israel Atomic Commission, Soreq Nuclear Research Center, Doar Yavne) 20-22. British **Biophysical** Soc., 20th

winter meeting, London, England. (R. E. Burge, Physics Dept., Queen Elizabeth College, Campden Hill Rd., London W.8) 20-22. American Physical Soc., Los

Angeles, Calif. (W. Whaling, California Inst. of Technology, Pasadena 91109)

